



# Luna: What Did We Learn and What Should We Expect?

William T. Wallace, Ph.D.  
Universities Space Research Association  
NASA/Johnson Space Center  
Houston, TX





# Outline

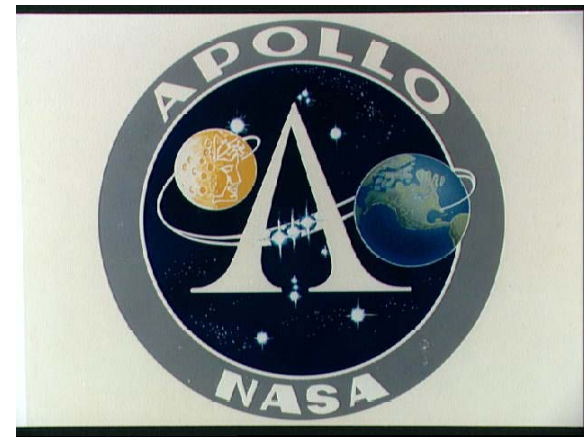
---

- Background
  - Apollo program
  - Lunar dust info
- Activation and monitoring
- Dissolution
- Cellular toxicology



# Kennedy Moon Speech

- Joint Session of Congress: May 25, 1961
- “First, I believe that this nation should commit itself to achieving the goal, before this decade is out, of landing a man on the moon and returning him safely to the earth.”
- Only 20 days after first U.S. manned space flight!





# Before the Moon



- Apollo 7 – Command module tests in low Earth orbit
- Apollo 8 – Command module to lunar orbit
- Apollo 9 – Lunar module tests in low Earth orbit
- Apollo 10 – Lunar module test in lunar orbit (down to ~ 9 miles above surface)







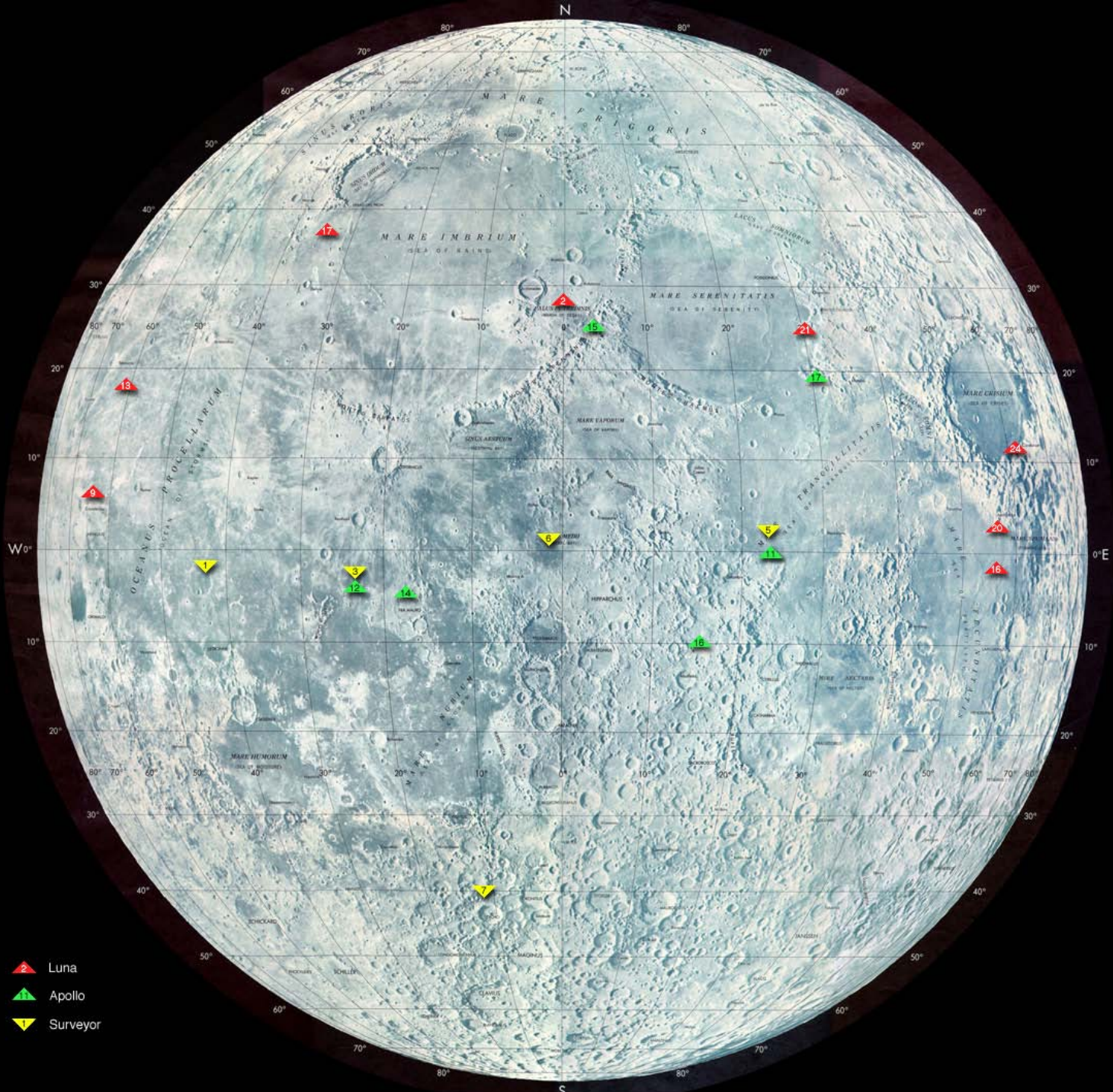


# Apollo Landings

	Mission Commander	Lunar Module Pilot	Command Module Pilot	Launch	Landing Site
Apollo 11	Neil Armstrong	Buzz Aldrin	Michael Collins	07/16/69	Sea of Tranquility
Apollo 12	Pete Conrad	Alan Bean	Dick Gordon	11/14/69	Ocean of Storms
Apollo 13	Jim Lovell	Fred Haise	Jack Swigert	04/11/70	Fra Mauro*
Apollo 14	Al Shepard (first lunar golfer)	Ed Mitchell	Stu Roosa	01/31/71	Fra Mauro
Apollo 15	Dave Scott	Jim Irwin	Al Worden	07/26/71	Hadley-Apennine
Apollo 16	John Young	Charlie Duke	Tom Mattingly	04/16/72	Descartes Highlands
Apollo 17	Gene Cernan	Jack Schmitt	Ron Evans	12/07/72	Taurus-Littrow

\*: Planned





-  Luna
-  Apollo
-  Surveyor





# Vision for Space Exploration

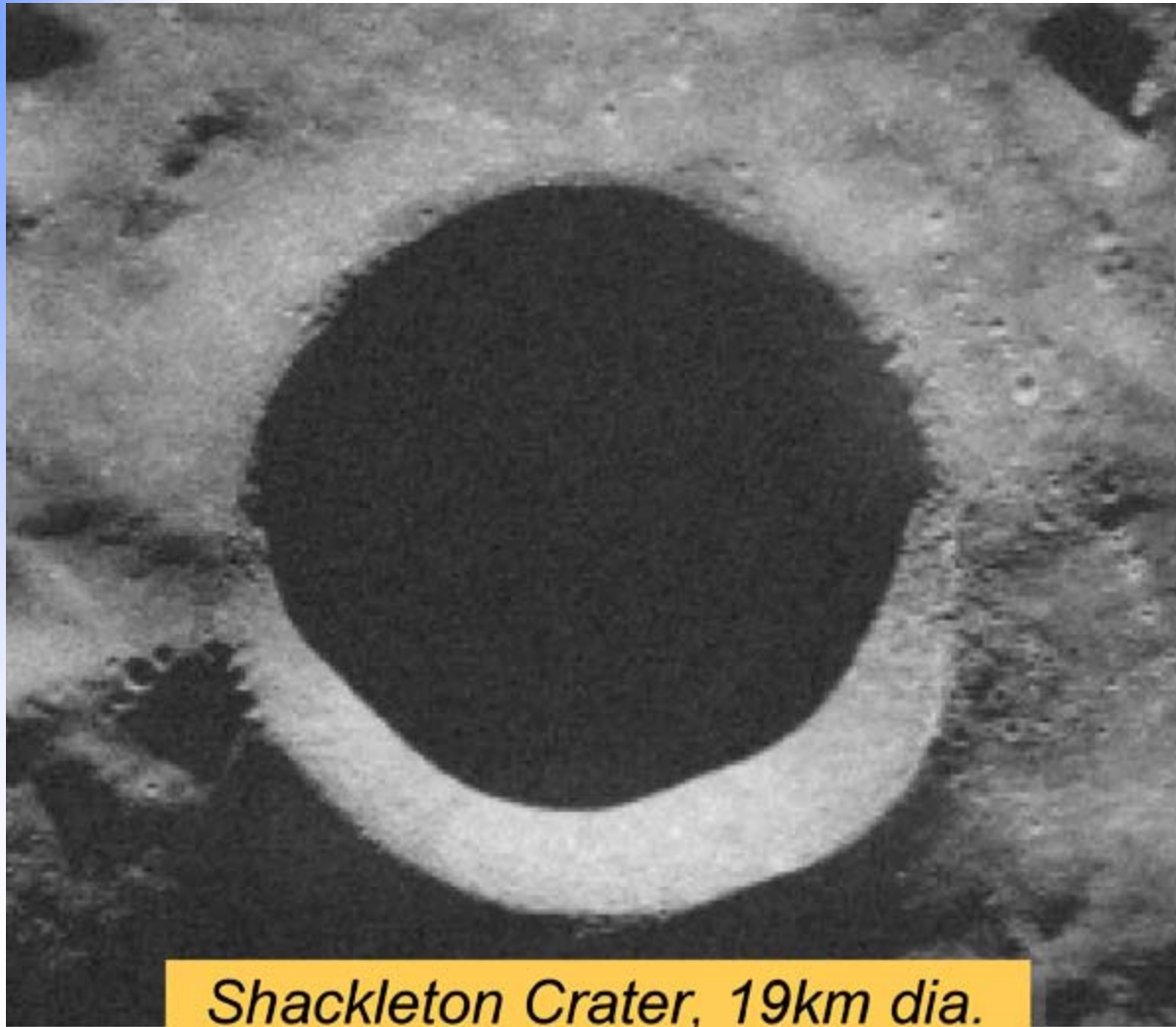
“With the experience and knowledge gained on the moon, we will then be ready to take the next steps of space exploration: human missions to Mars and to worlds beyond.”

- President George W. Bush  
(January 14, 2004)





# Possible Outpost Site



*Shackleton Crater, 19km dia.  
(lunar South Pole)  
2005 Arecibo radar image*

## Why the South Pole?

- Safe
  - Thermally moderate
- Cost Effective
  - High % of sunlight
  - Allows the use of solar power
- Resources
  - Enhanced hydrogen (possibly H<sub>2</sub>O)
  - Potentially other volatiles
  - Oxygen
- Flexibility
  - Allows incremental buildup using solar power
  - Enhanced surface daylight
  - More opportunities to launch
- Exciting
  - Not as well known as other areas
  - Offer unique, cold, dark craters





# Words of Wisdom

*“I think dust is probably one of our greatest inhibitors to a nominal operation on the Moon. I think we can overcome other physiological or physical or mechanical problems except dust.”*

*Gene Cernan  
Apollo 17 Technical  
Debrief*





# Lunar EVA Suits



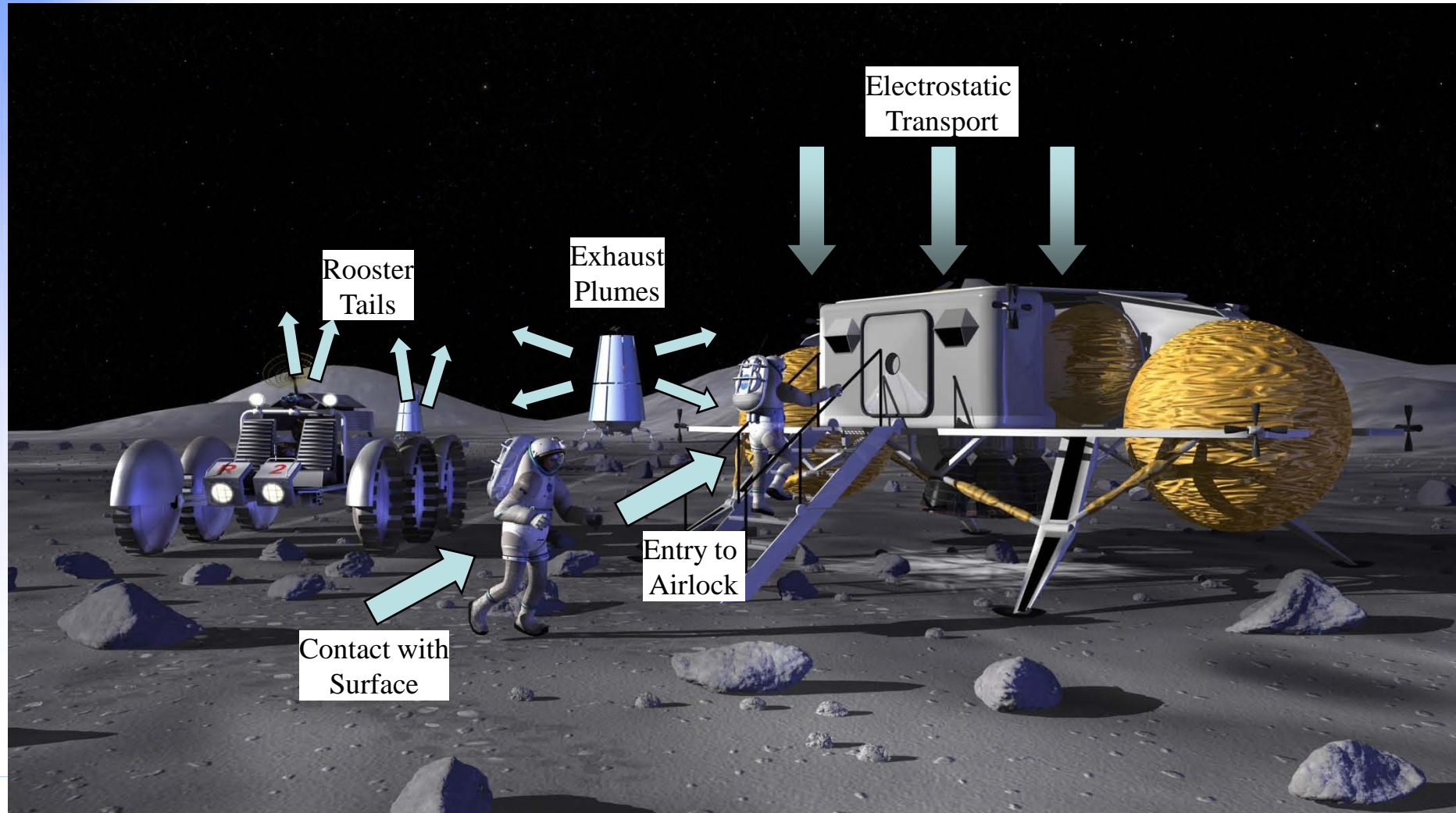
Jack Schmitt  
(Apollo 17)







# Dust Exposure Possibilities





# Problems Caused by Dust

- Obscured vision
  - Apollo 15: vision completely obscured below 60 ft when landing
- Clogged equipment
  - Apollo 12: wrist and suit hose locks clogged with dust
- Coated surfaces
  - Apollo 11: T.V. cable caused tripping after dust covering
- Inhalation
  - Apollo 15: gunpowder smell
  - Apollo 17: “hay fever” symptoms
- Degraded radiators
  - Apollo 16: degraded instrument performance from overheating
- Fooled instruments
- Caused seal failure
  - Apollo 14: measurable leaking of suits
- Abraded surfaces
  - Apollo 16: gauge dials unreadable from scratching





# What is lunar dust?

---

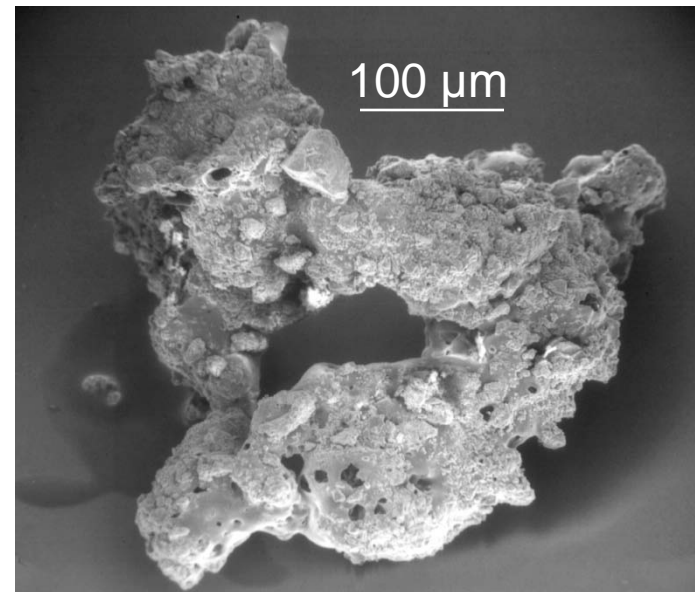
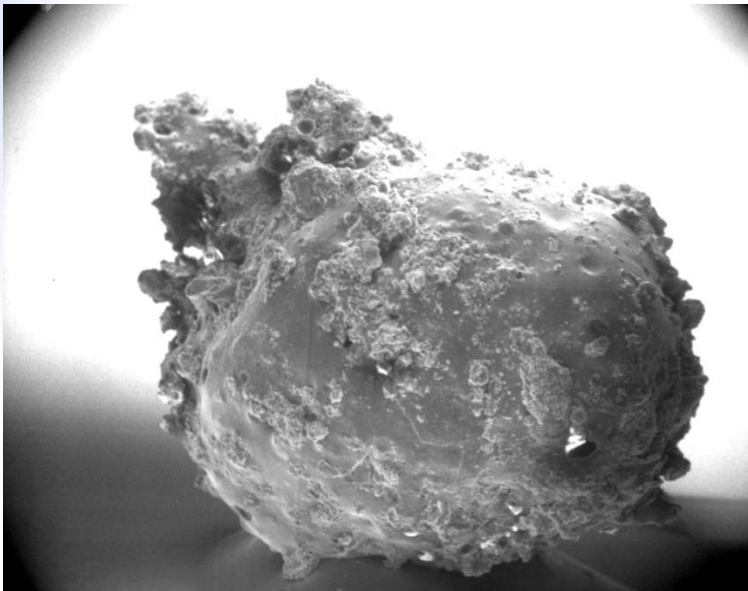
- **Lunar soil** is defined as the loose fragmental material with a grain size smaller than 1 cm on and near the surface of the moon. It is a subset of the lunar regolith which includes all size fragments including boulders.
- **Lunar dust** is the finest size fraction of lunar soil. A working definition of lunar dust is that it is all grains smaller than 20  $\mu\text{m}$ .



# Lunar Dust

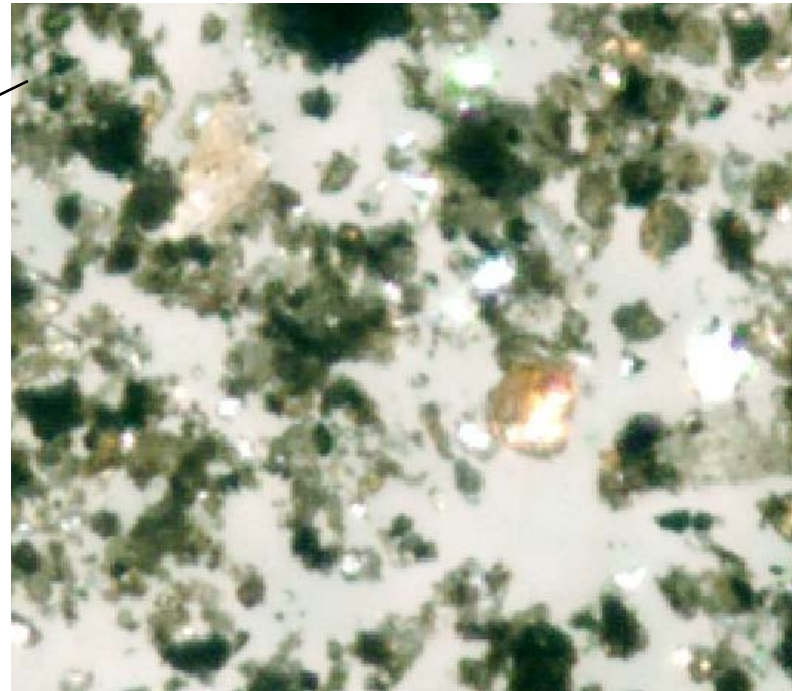
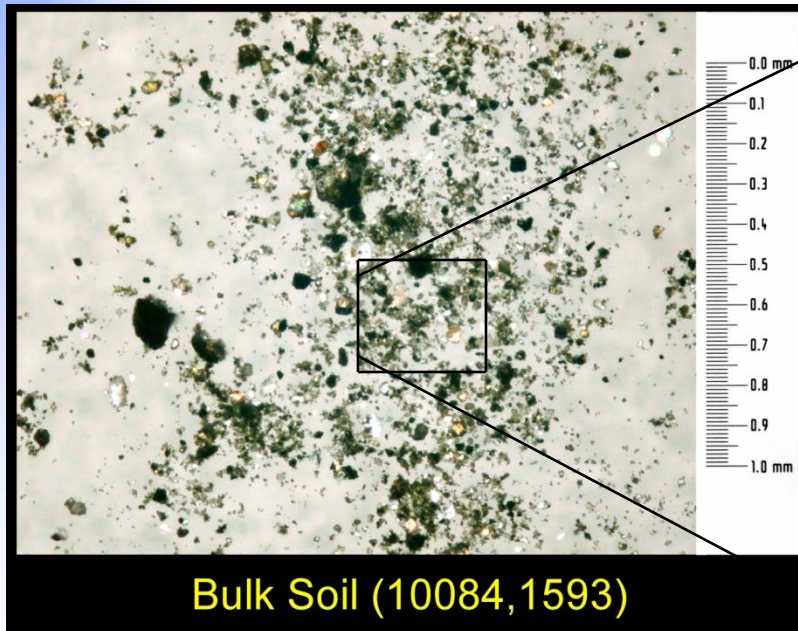


- Contains  $\text{SiO}_2$ , other oxides, and trace metals
- Magnetic
- Particles are oddly shaped, with jagged edges, and do not pack together well





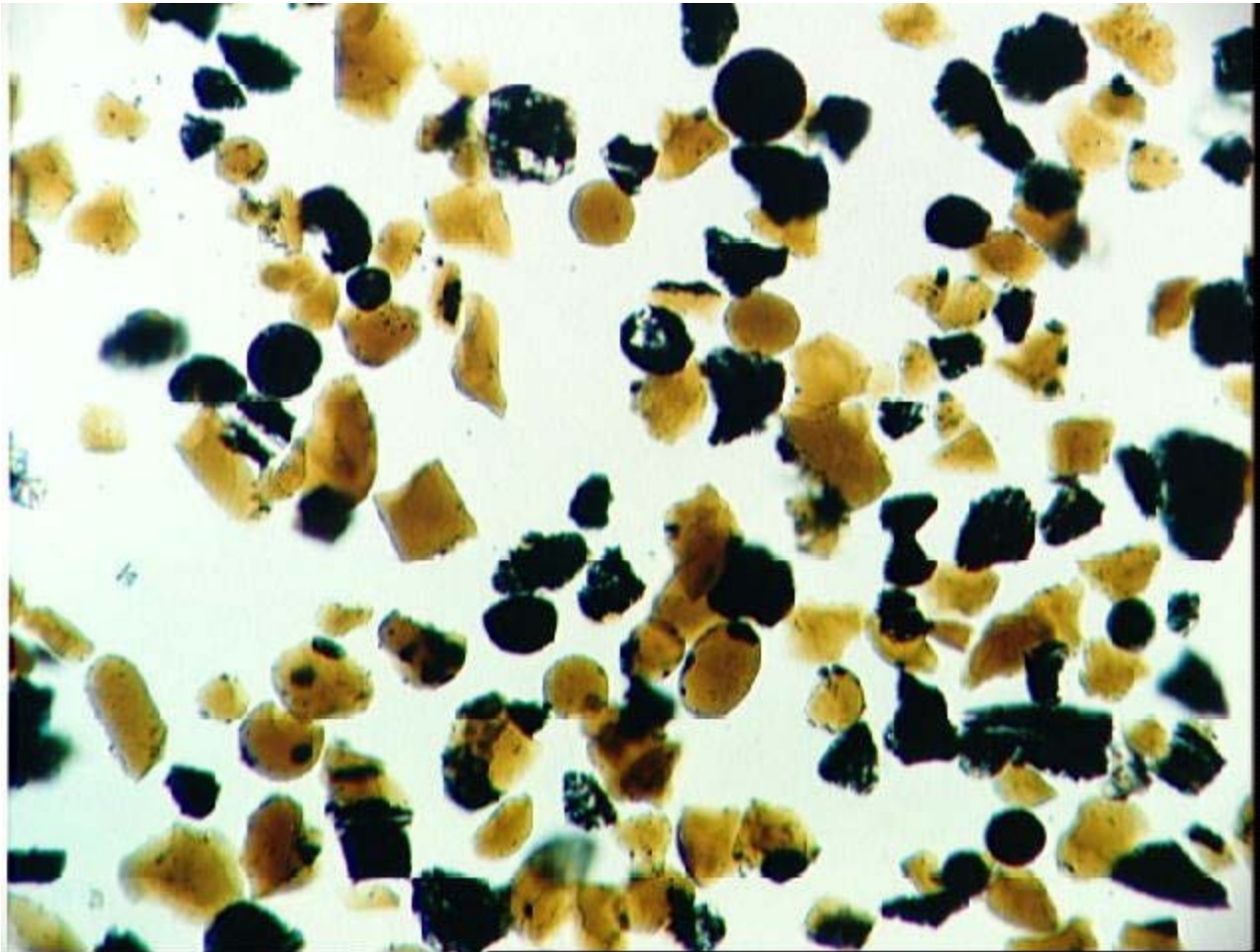
# Bulk Apollo 11 Soil



20  $\mu\text{m}$



# Orange Soil



- From Taurus-Littrow (Apollo 17)
- 25-45  $\mu\text{m}$
- Produced by volcanic eruption of pyroclastic ash



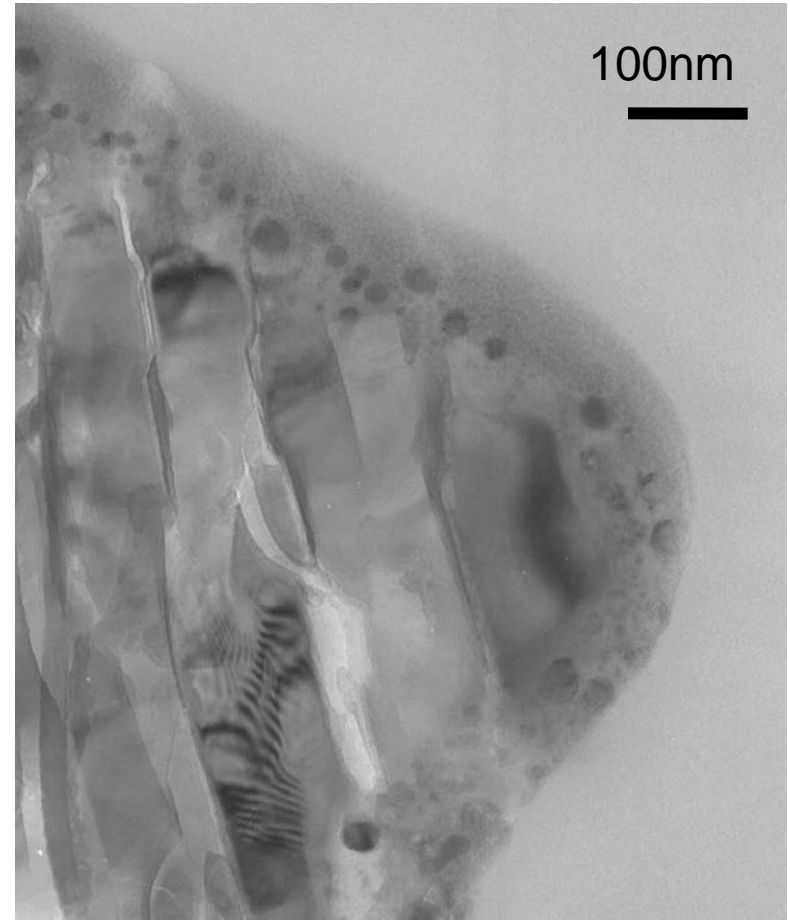
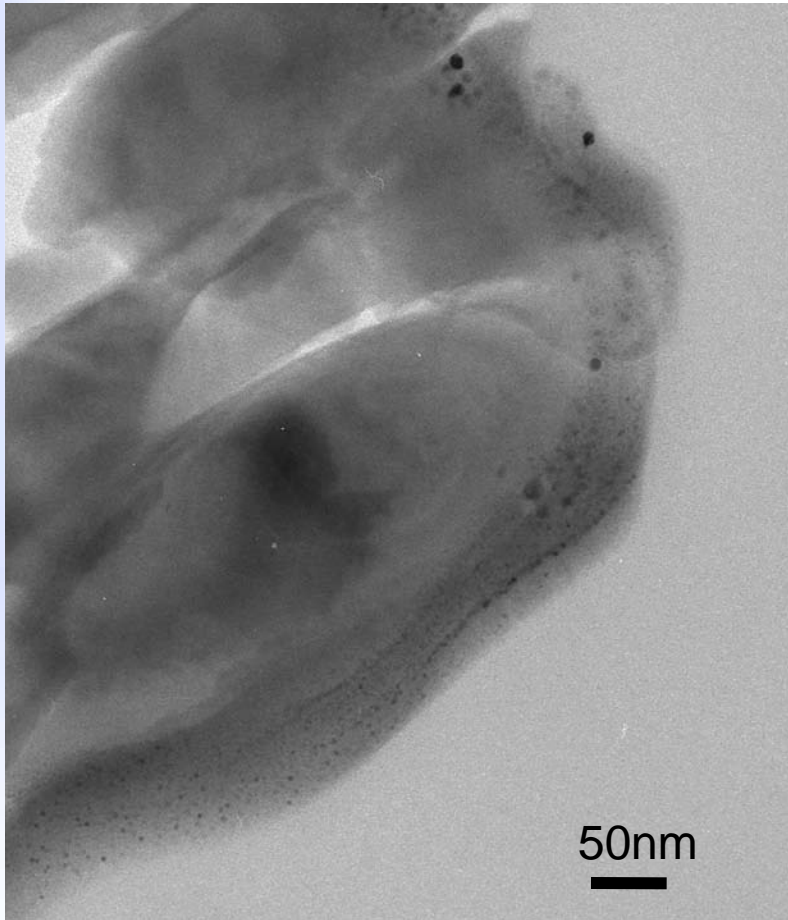
# Green Soil

- Apollo 15
- Formed from same type of process as Apollo 17 orange soil





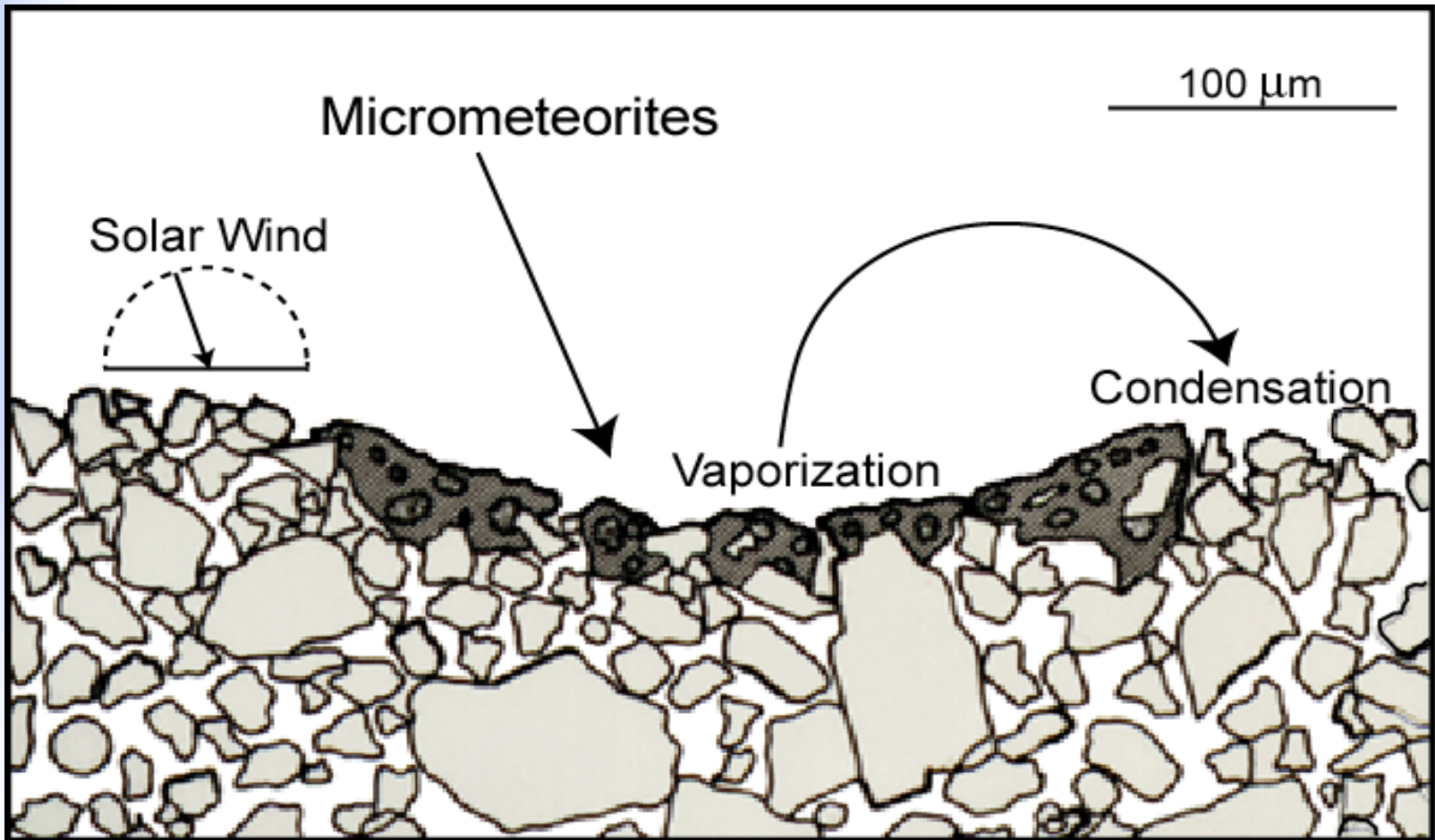
# Lunar Dust Rims



Glassy rims produced by vapor/sputter deposition. Also contain ~ 10 nm Fe nanoparticles



# Lunar Soil Formation



Lunar soil is formed by a combination of comminution (breaking down), agglutination (clumping together), and vapor deposition.



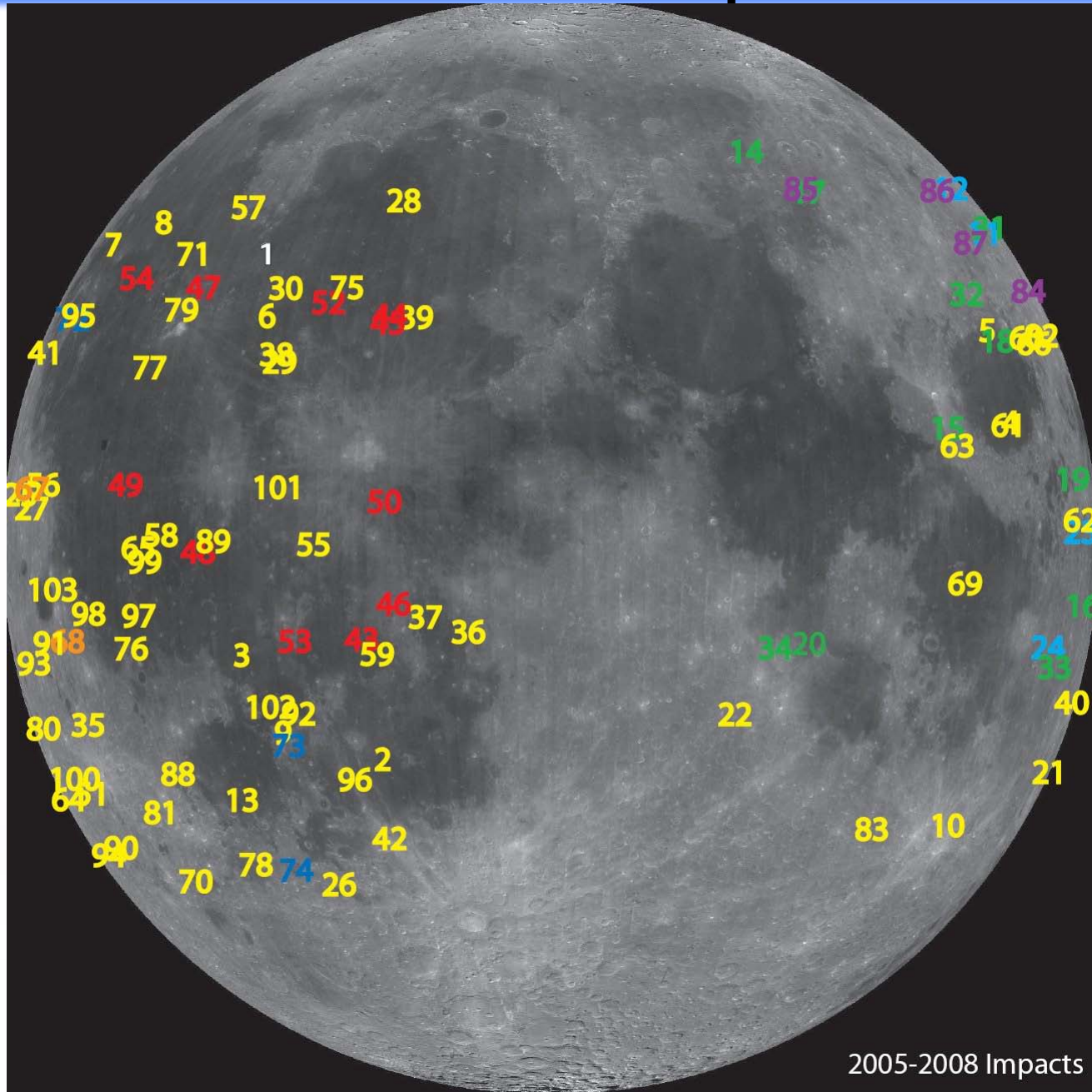
# Meteorite Impact on the Moon



- 25 cm diameter meteorite traveling at 85,000 mph
- Kinetic energy of impact: 17 billion joules (equivalent to 4 tons of TNT)
- New crater: 14 meters wide by 3 meters deep
- Flash only 0.4 seconds in real-time



# Recent Impacts

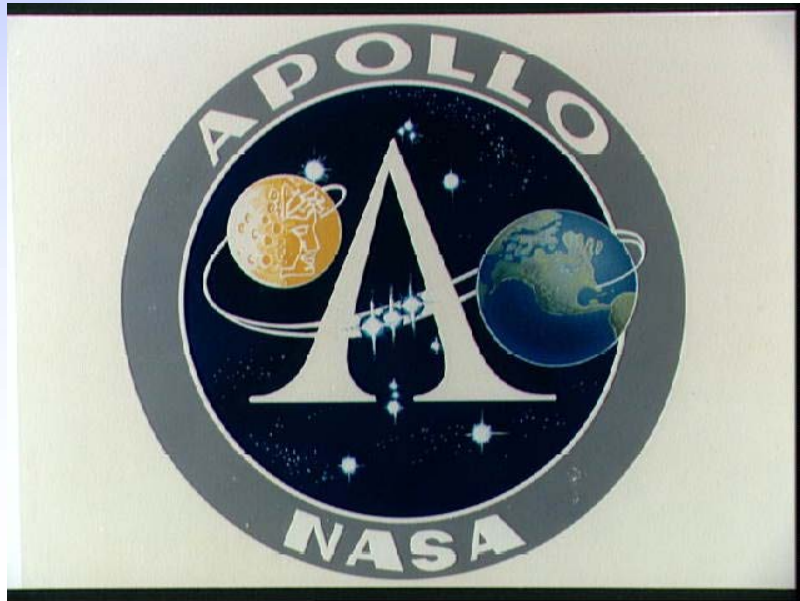






# Further Info on Apollo

- <http://www.hq.nasa.gov/alsj/main.html>  
(Apollo Lunar Surface Journal)





# Lunar Dust Simulant

Only 842 lbs of material returned from the moon!  
Simulant material needed for preliminary studies.

- JSC-1A-vf
- Made from volcanic ash
- 50% silicon-containing minerals
- 42-45% other oxides ( $\text{Al}_2\text{O}_3$ ,  $\text{FeO}$ ,  $\text{MgO}$ ,  $\text{CaO}$ )
- No trace metals
- Size distribution of particles similar to samples of lunar dust
- 90% smaller than 13  $\mu\text{m}$  diameter



# Materials Used

Sample	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	TiO <sub>2</sub>	FeO	MnO	MgO	CaO	Na <sub>2</sub> O	K <sub>2</sub> O	P <sub>2</sub> O <sub>5</sub>
JSC-1A-vf, % oxides	48.77	15.65	1.49	8.88 (+ 1.71% Fe <sub>2</sub> O <sub>3</sub> )	0.19	8.48	10.44	2.93	0.81	0.66
Apollo 16 Soil (62241), % oxides	44.65	27	0.56	5.49	0.7	5.84	15.95	0.44	0.13	0.1
Min-U-Sil Quartz, %	99.0- 99.9	< 0.8	< 0.1	< 0.1 (Fe <sub>2</sub> O <sub>3</sub> )	0	0	0	0	0	0





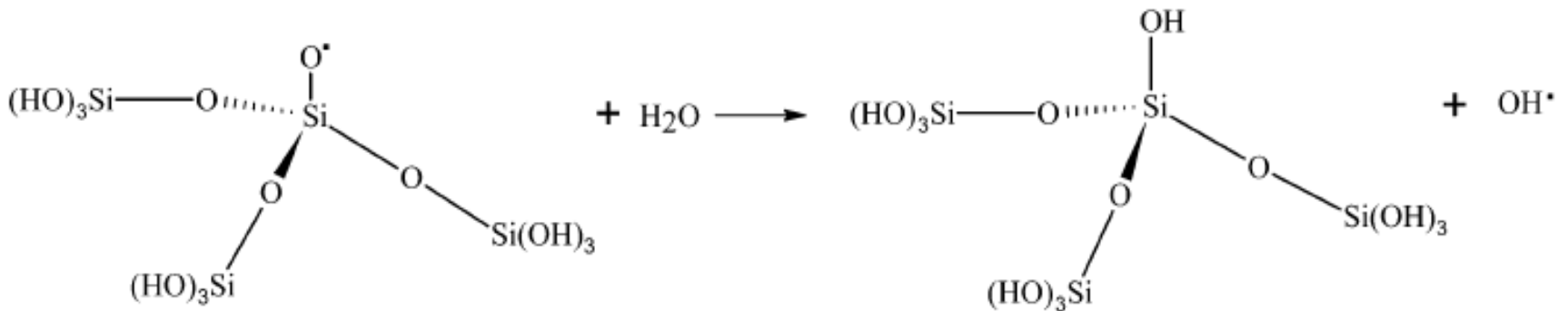
# Lunar Dust Activation

- Constant activation of lunar dust by meteorites, UV radiation, and elements of solar wind
- No passivating atmosphere
- Active dust could produce reactive species in the lungs
  - Freshly fractured quartz
- Must determine methods of deactivation before new lunar missions
- First, must understand how to *reactivate* dust on Earth



# What Does “Activated” Mean?

- Presence of reactive sites on surface
  - Free radicals
- Ability to produce reactive species in solution



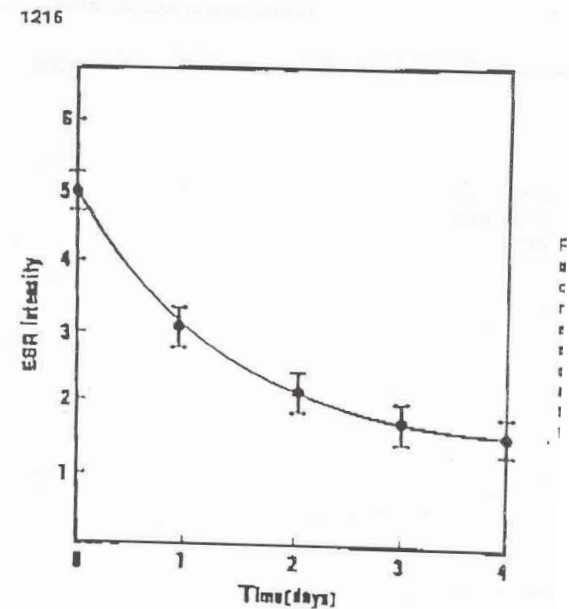
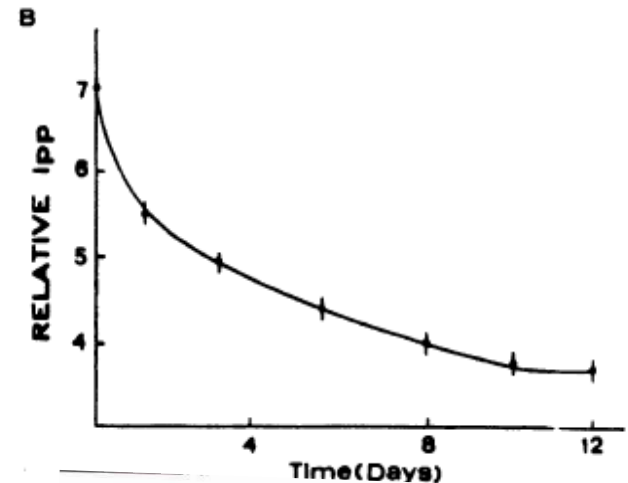
Reaction 5



# Previous Studies of Quartz Activation

- Grinding quartz gives electron paramagnetic resonance (EPR) signal characteristic of Si· or Si-O· radicals
- Increased grinding time produces higher signal
- Decrease in Si-based radicals over time in air
  - Half-life of ~30 hours, with 20% of signal detectable even after 4 weeks

- Ground quartz in aqueous solution produces OH radicals
- Radical production decreases with exposure to air
  - Half life of ~ 20 hours







# Activation Methods Tested

---

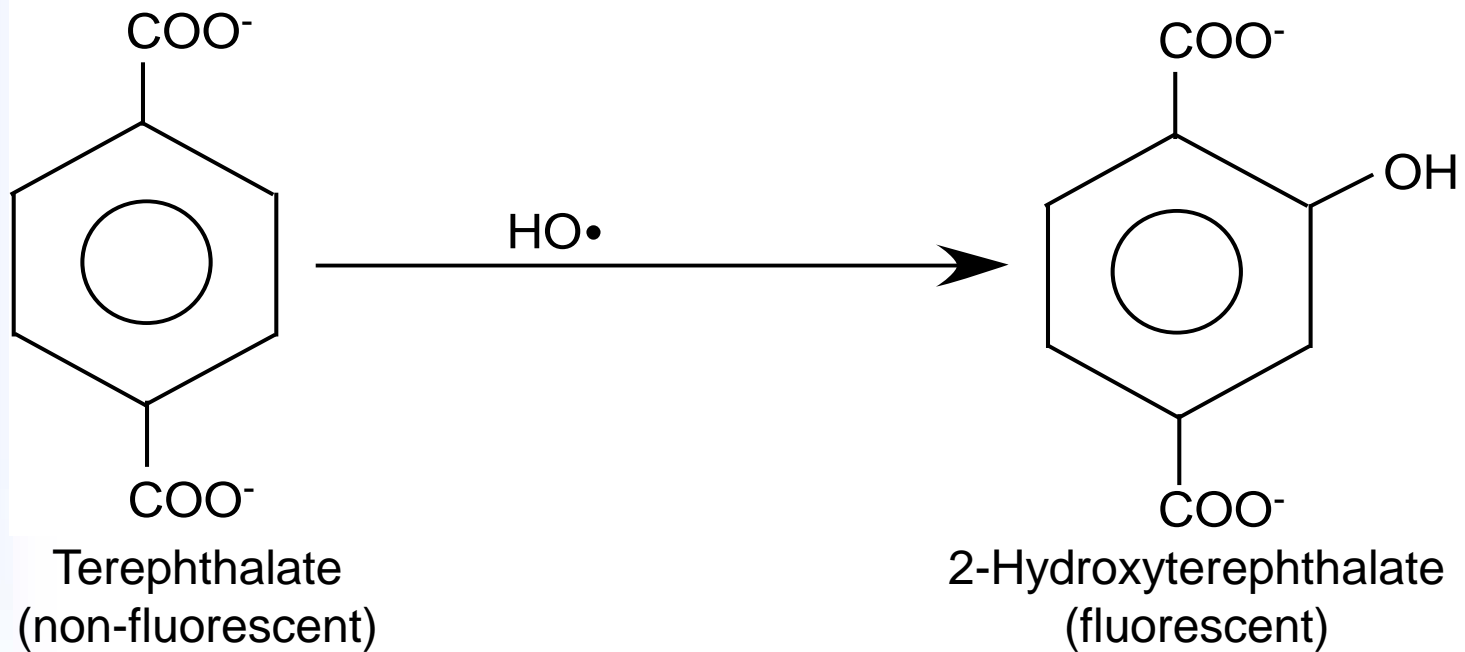
- Crushing/Grinding
  - Mortar and pestle
  - Ball Mill
- UV activation



# Fluorescence



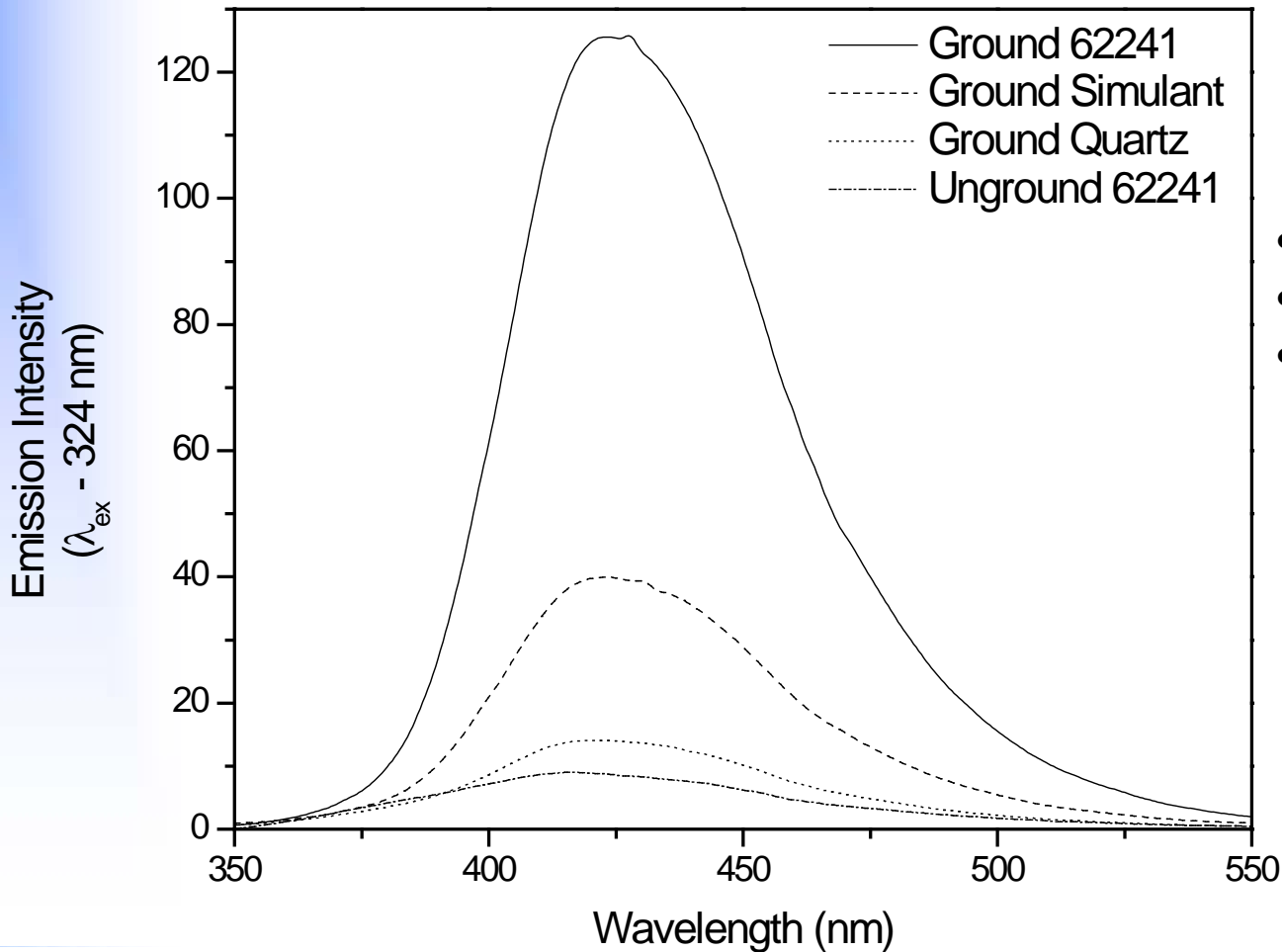
# Hydroxyterephthalate as a Probe of Hydroxyl Radical Generation







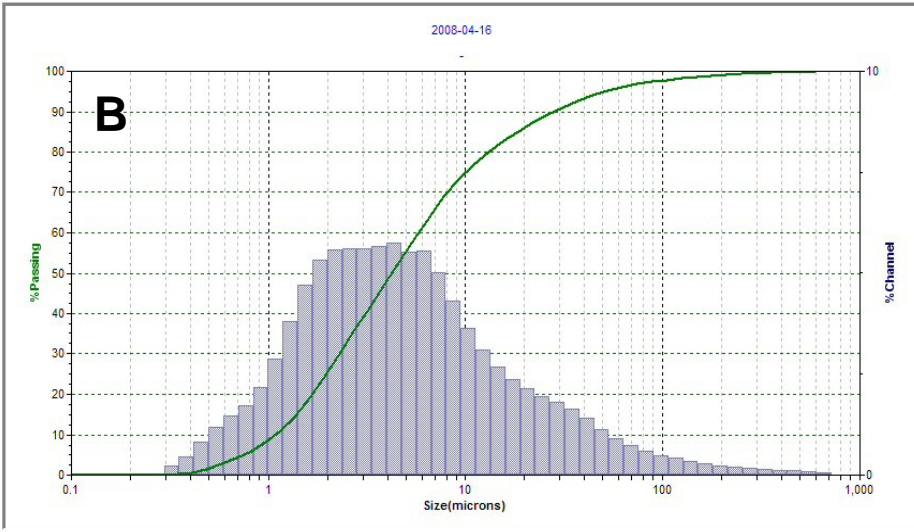
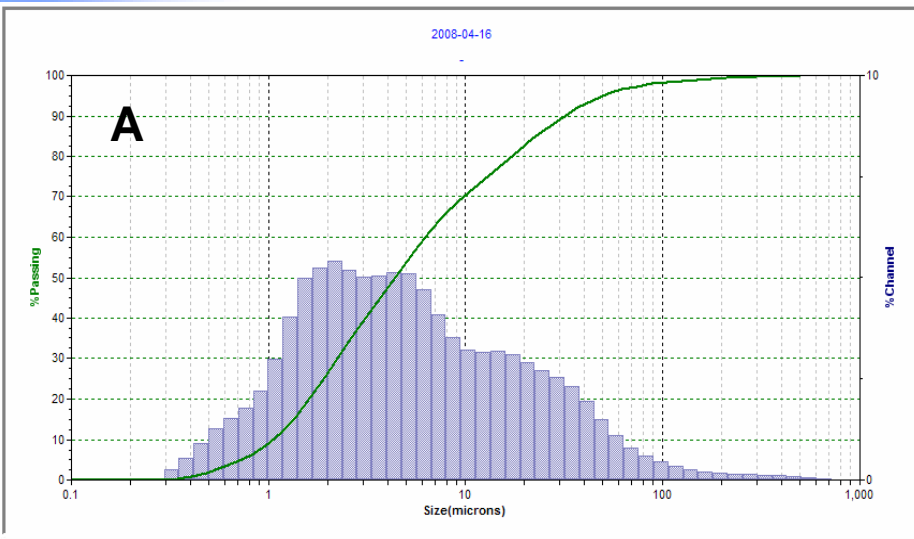
# Activity Comparison of Ground Lunar Soil, Lunar Simulant, and Quartz



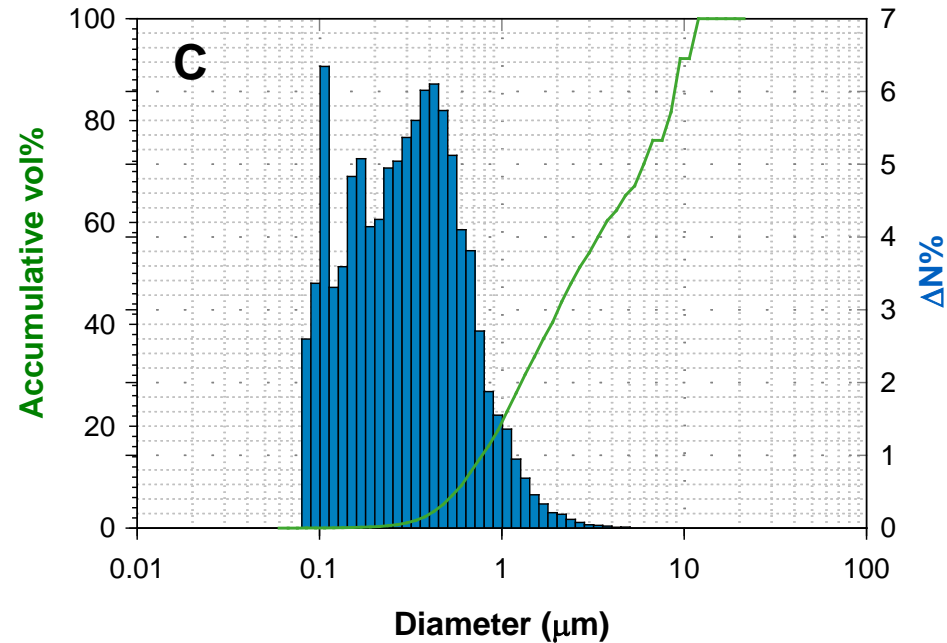
- 10 minute grinding
- 3.8 mg/mL JSC-1A-vf
- 10 mM Terephthalate



# Size Distribution and Surface Area after Grinding



Lunar dust particles (62241-Ground)



**A: Min-U-Sil 15- 8.436 m<sup>2</sup>/g**  
(unground- 3.7 m<sup>2</sup>/g)

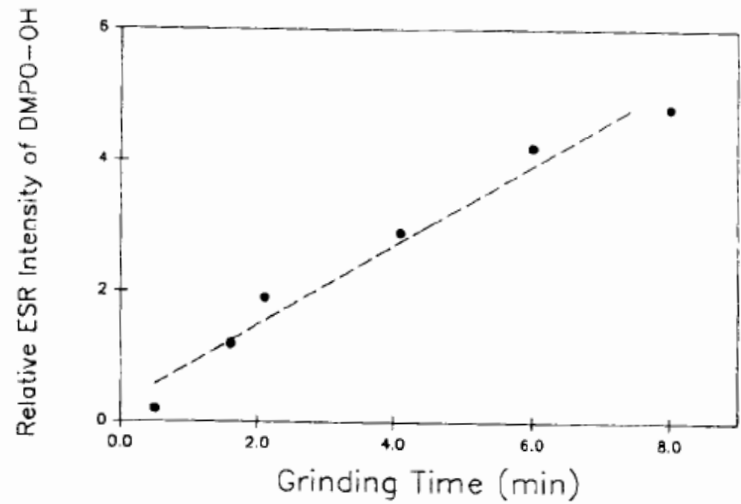
**B: JSC-1A-vf- 5.369 m<sup>2</sup>/g**  
(unground- 3.5 m<sup>2</sup>/g)

**C: 62241 (Apollo 16)- 8.404 m<sup>2</sup>/g**  
(unground- 1.6 m<sup>2</sup>/g)

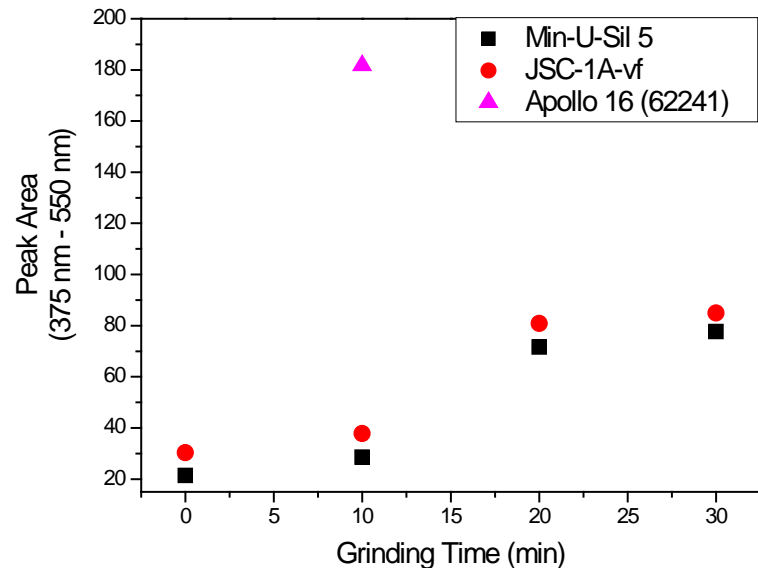


# Effect of Grinding Time

- Grinding time has a direct effect on amount of hydroxyl radicals produced upon addition of ground quartz to solution
- Grinding time also shown to produce higher number of silicon-based radicals in ESR spectra
- Increase in hydroxyl production also seen for lunar simulant with increased grinding



Fluorescence Signal Produced by Grinding



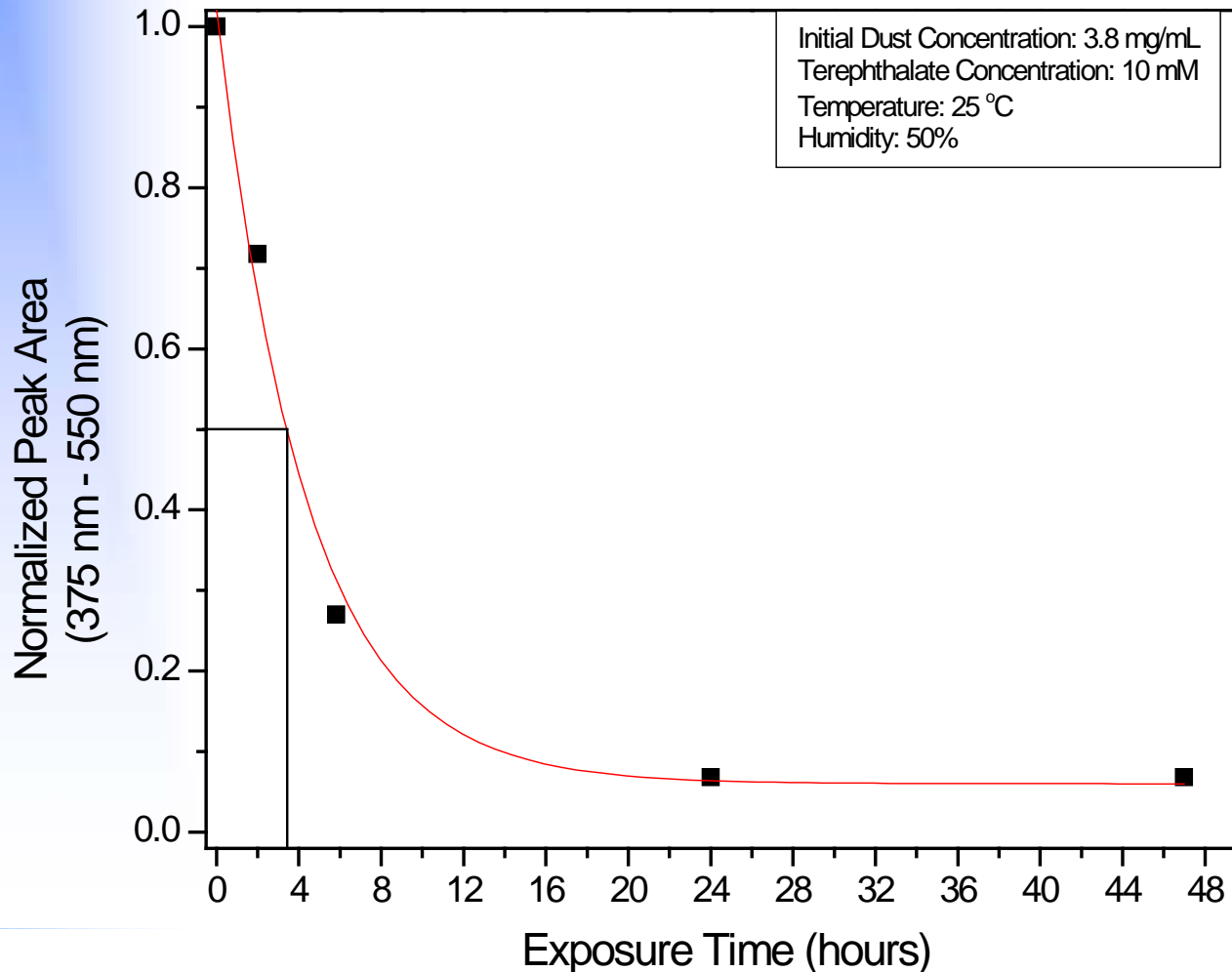




# Deactivation after Grinding



# Deactivation of Freshly Ground Lunar Simulant (JSC-1A-vf)

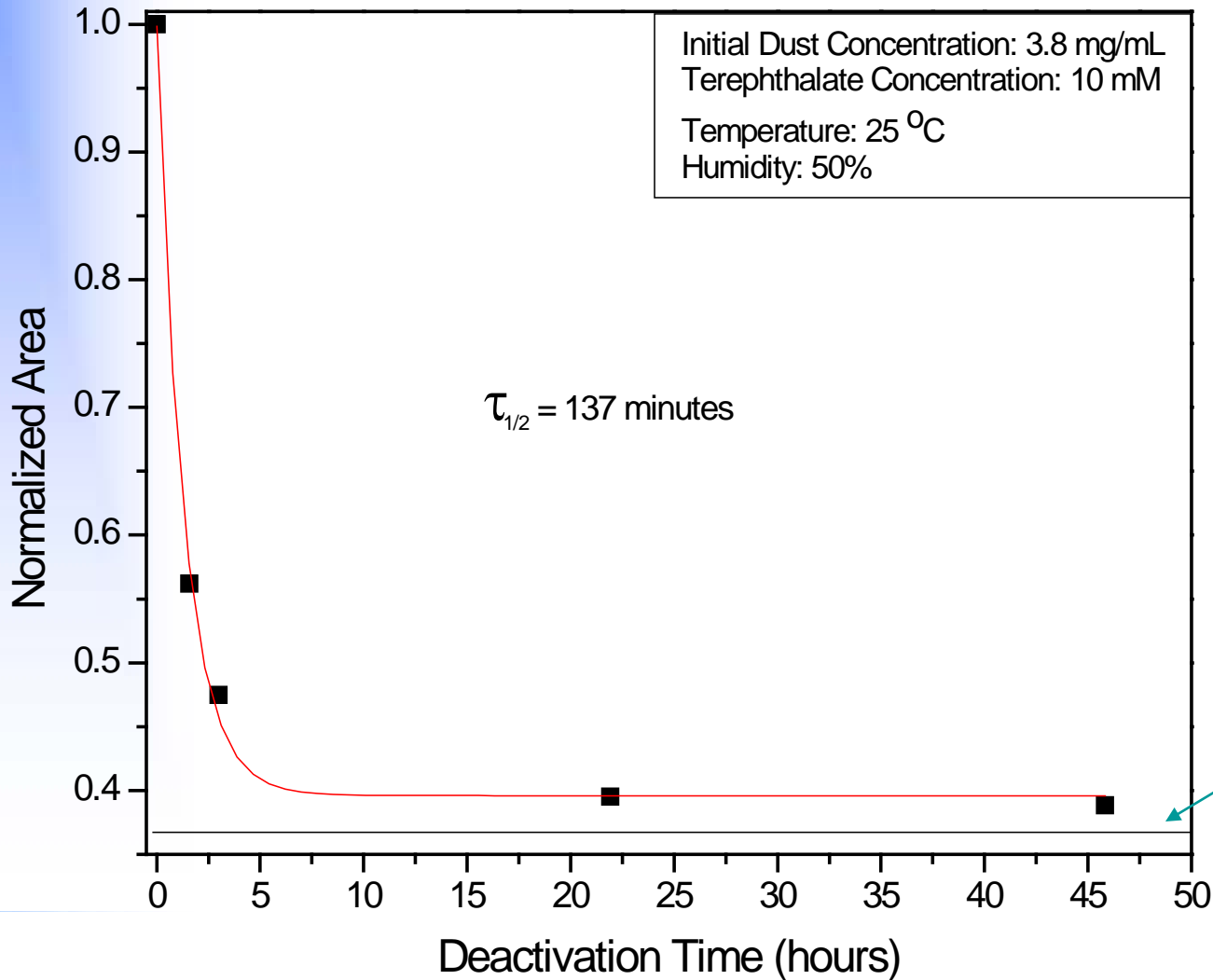


- Activity of freshly ground simulant can be reduced by exposure to humid environment.

- Multiple sets of deactivation experiments show simulant half life to be ~ 3 hours with activity approaching unground levels at ~ 24 hours.



# Deactivation of Freshly Ground Quartz



Much faster deactivation of quartz, especially with respect to approach of unground activity.

Activity of unground quartz (close to zero intensity; normalized to freshly ground)

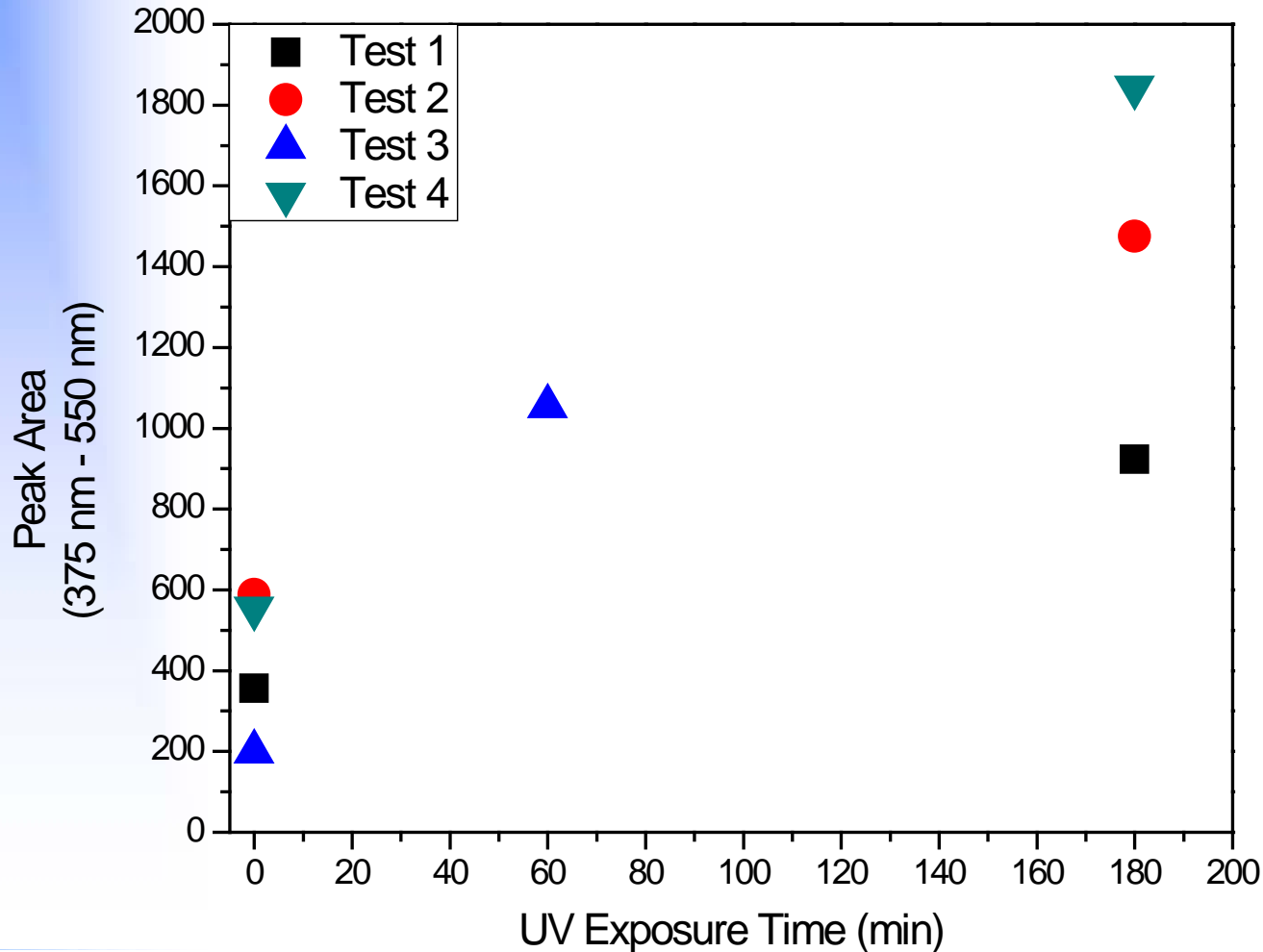




# Activation by UV Exposure and Heating



# UV Activation of Unground Lunar Simulant

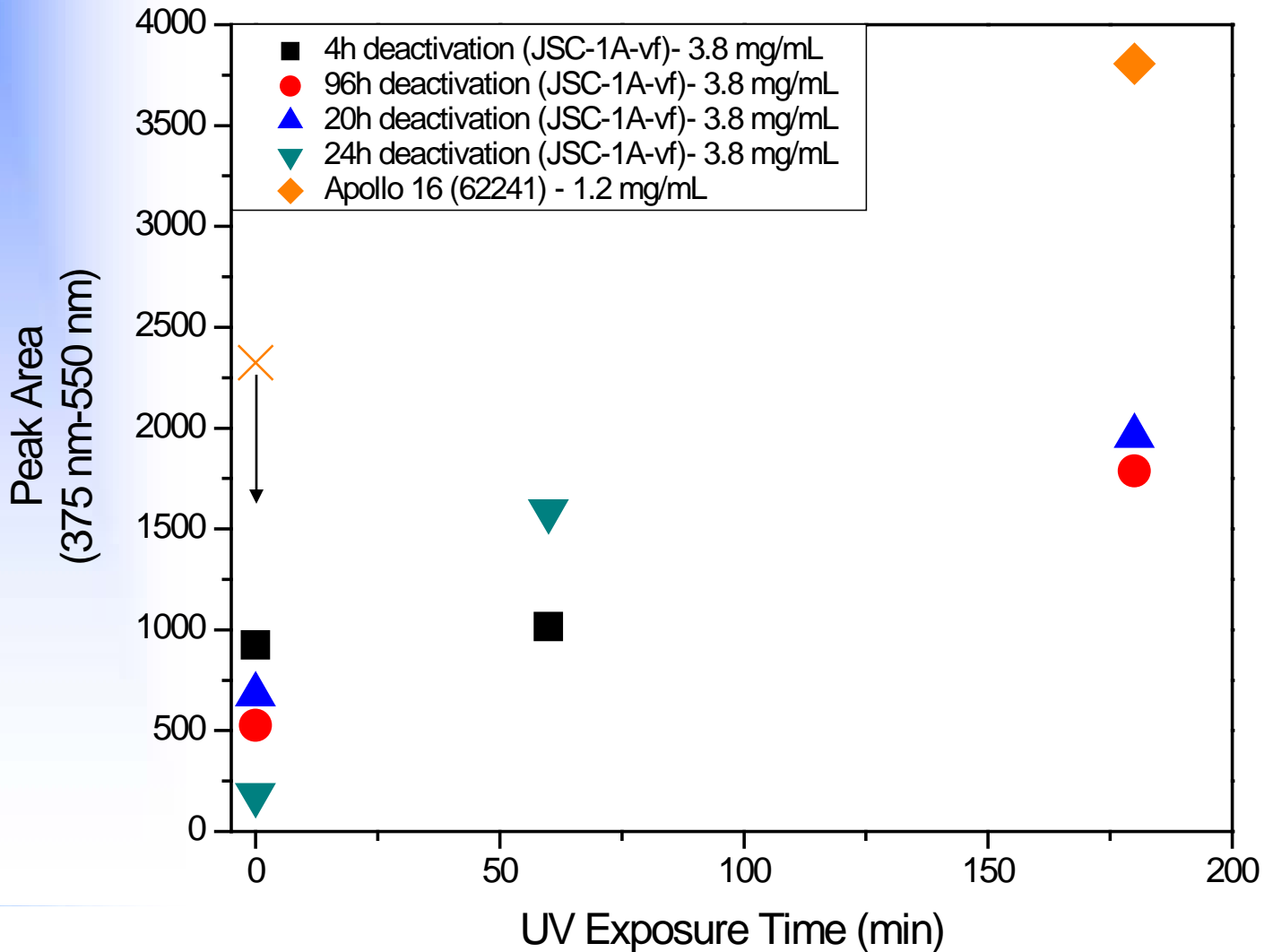


- 3.8 mg/mL JSC-1A-vf
- 10 mM Terephthalate
- 800 W UV (initial setting)
- $\sim 5 \times 10^{-4}$  Torr

Exposure of unground simulant to UV radiation under vacuum leads to the production of hydroxyl radicals by the simulant when placed in solution.



# UV Reactivation of Ground, Deactivated Lunar Dust and Simulant



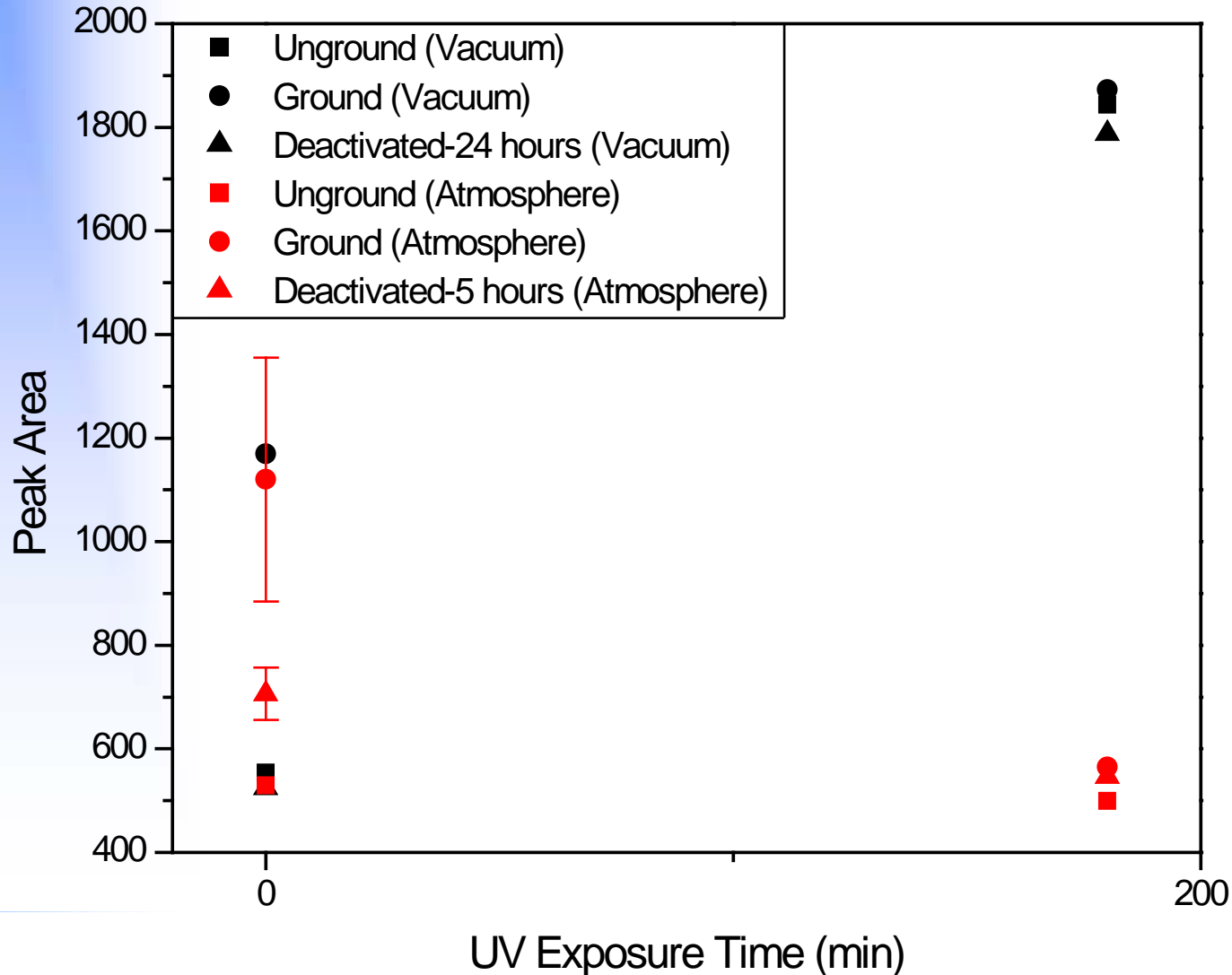
Final deactivation point from initial lunar dust testing (> 1 month further deactivation; upper limit for activity)

Deactivated (50% RH, 25 °C) lunar simulant and lunar dust can be reactivated by exposure to UV radiation under vacuum.



# Effects of Vacuum on UV

## Activation/Deactivation of Lunar Simulant



- 3.8 mg/mL
- 10 mM Terephthalate
- 800 W UV (initial setting)

- Error bars for deactivated and ground simulant account for activities prior to and at conclusion of UV exposure.

Exposure of active simulant to UV in air leads to deactivation!

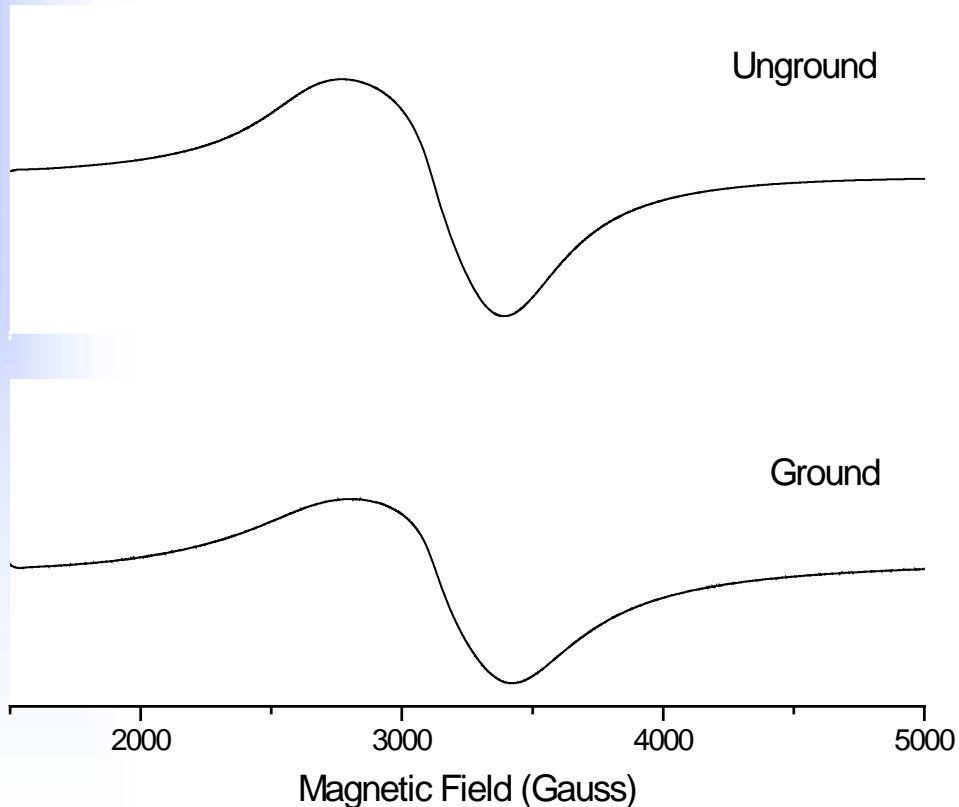




# EPR



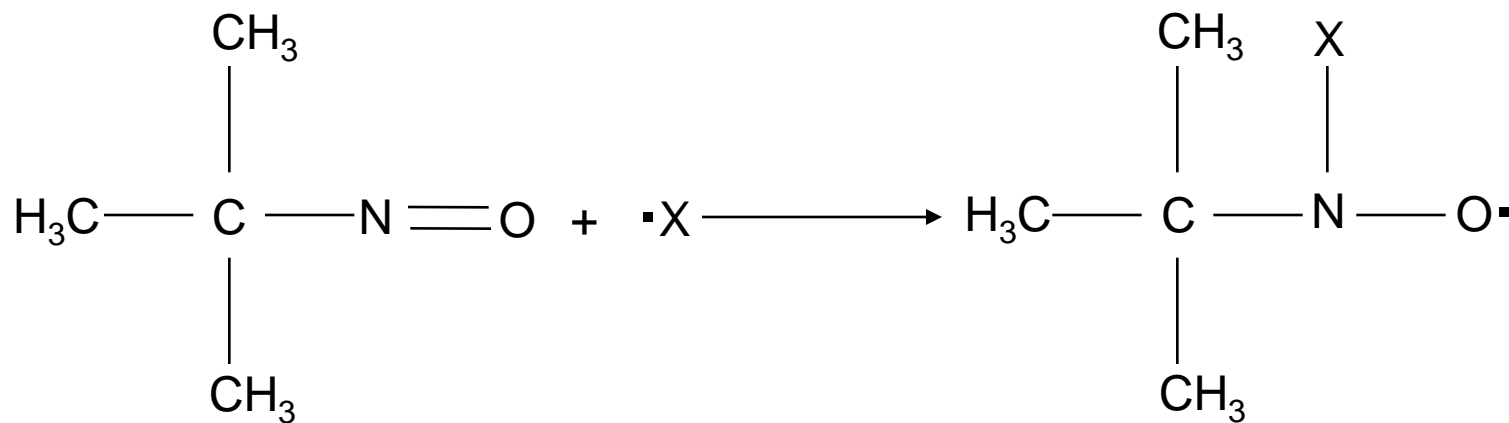
# EPR Spectra of Apollo 62241



- Broad peaks: no determination of silicon- or oxygen-based radicals
- Change in g-values from 2.11 (unground) to 2.09 (ground)
- Similar downward shifts and g-values seen previously by Haneman and Miller\*

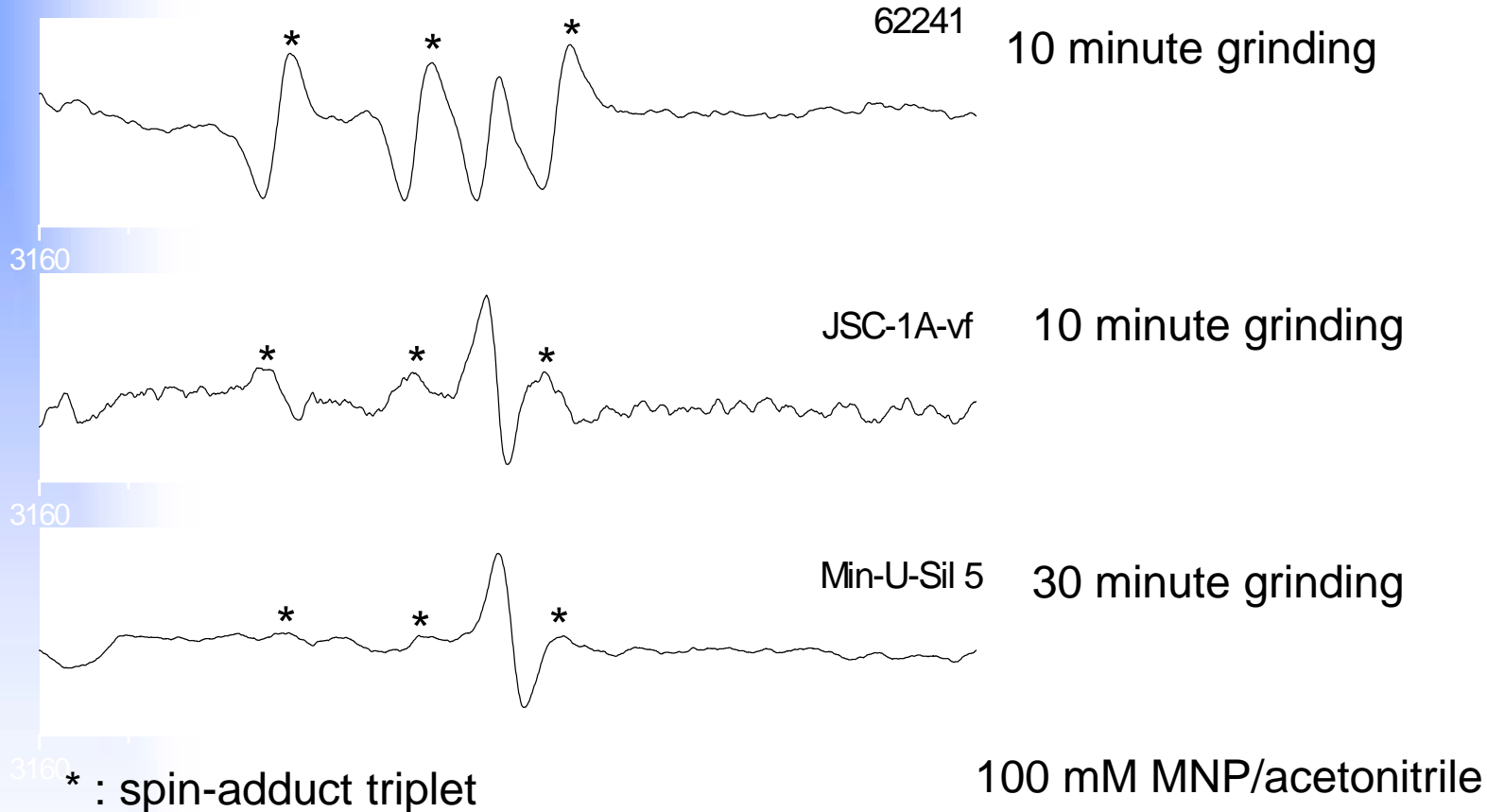


# MNP Spin-adduct Reaction





# Spin-trapping of Radicals



- Level of activity increases in the order: quartz < lunar dust simulant < lunar dust
- Peak-to-peak splitting corresponds to radical containing no hydrogen
  - Activated species likely interacting with acetonitrile to produce radicals
- Future testing to include hydroxyl radical trap in water





# Solubility



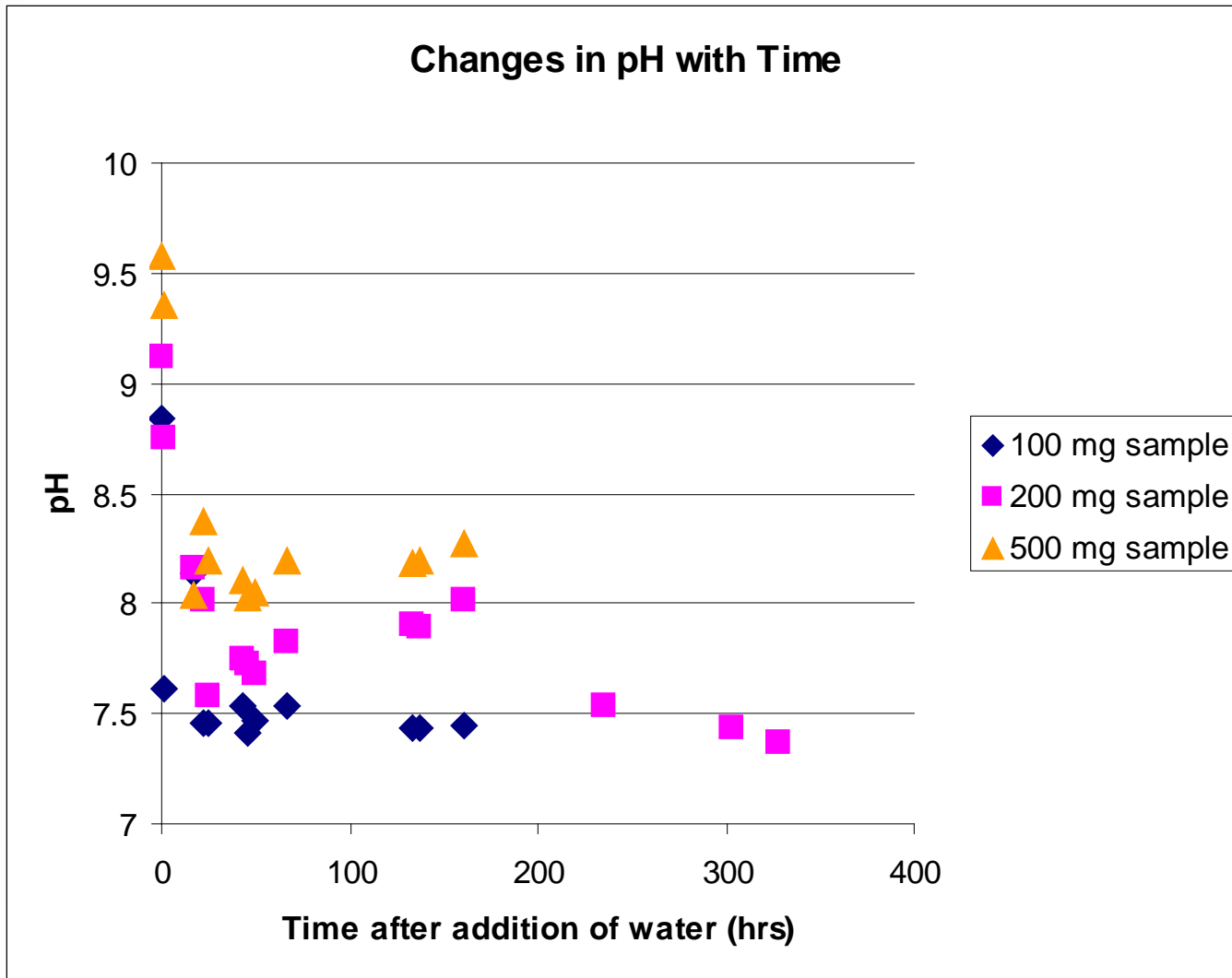
# Technique

---

- Place 10 mg JSC-1A-vf in 20 mL of buffer solution in 50 mL centrifuge tube
- Rotate tubes for 72 hours under ambient conditions (23-25 °C, 30-50% RH)
- Flush syringes and 0.2  $\mu\text{m}$  syringe filters with distilled water
- Filter solutions
- Measure concentrations using ICP-MS



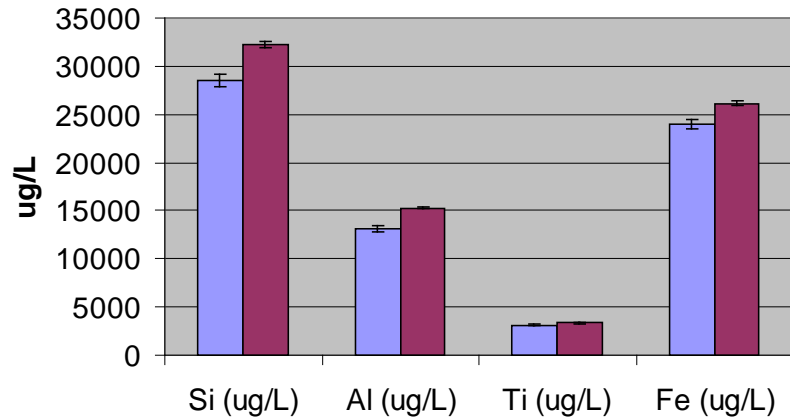
# pH Effects



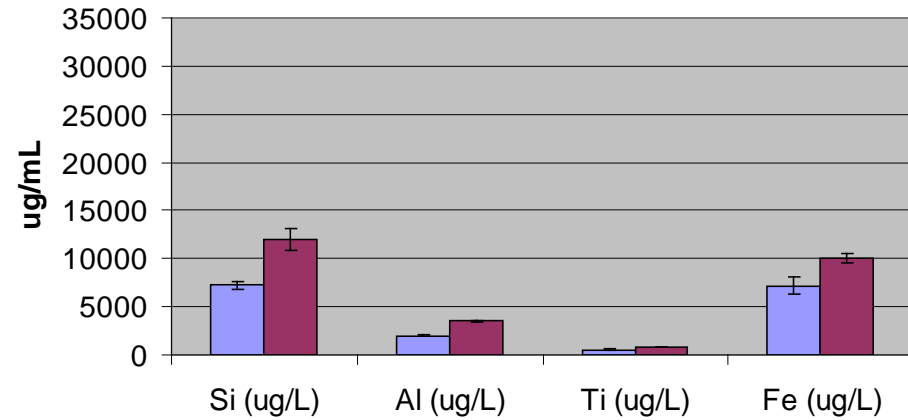


# Effect of pH on Leaching

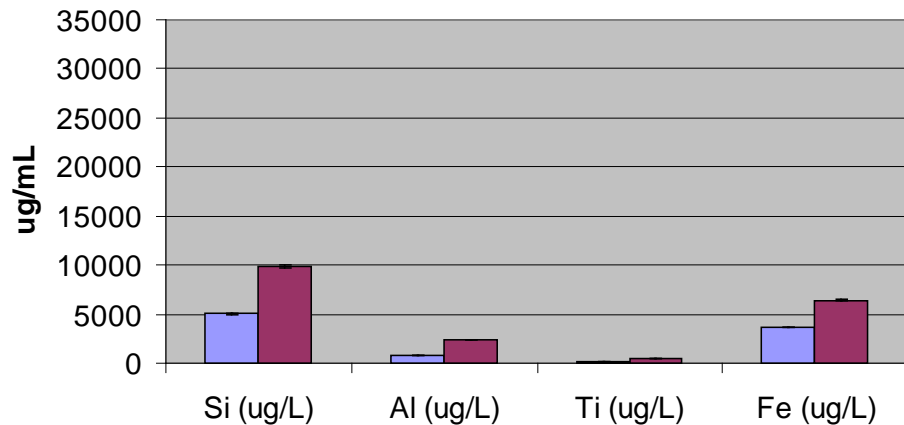
pH 4.0



pH 5.3



pH 6.7

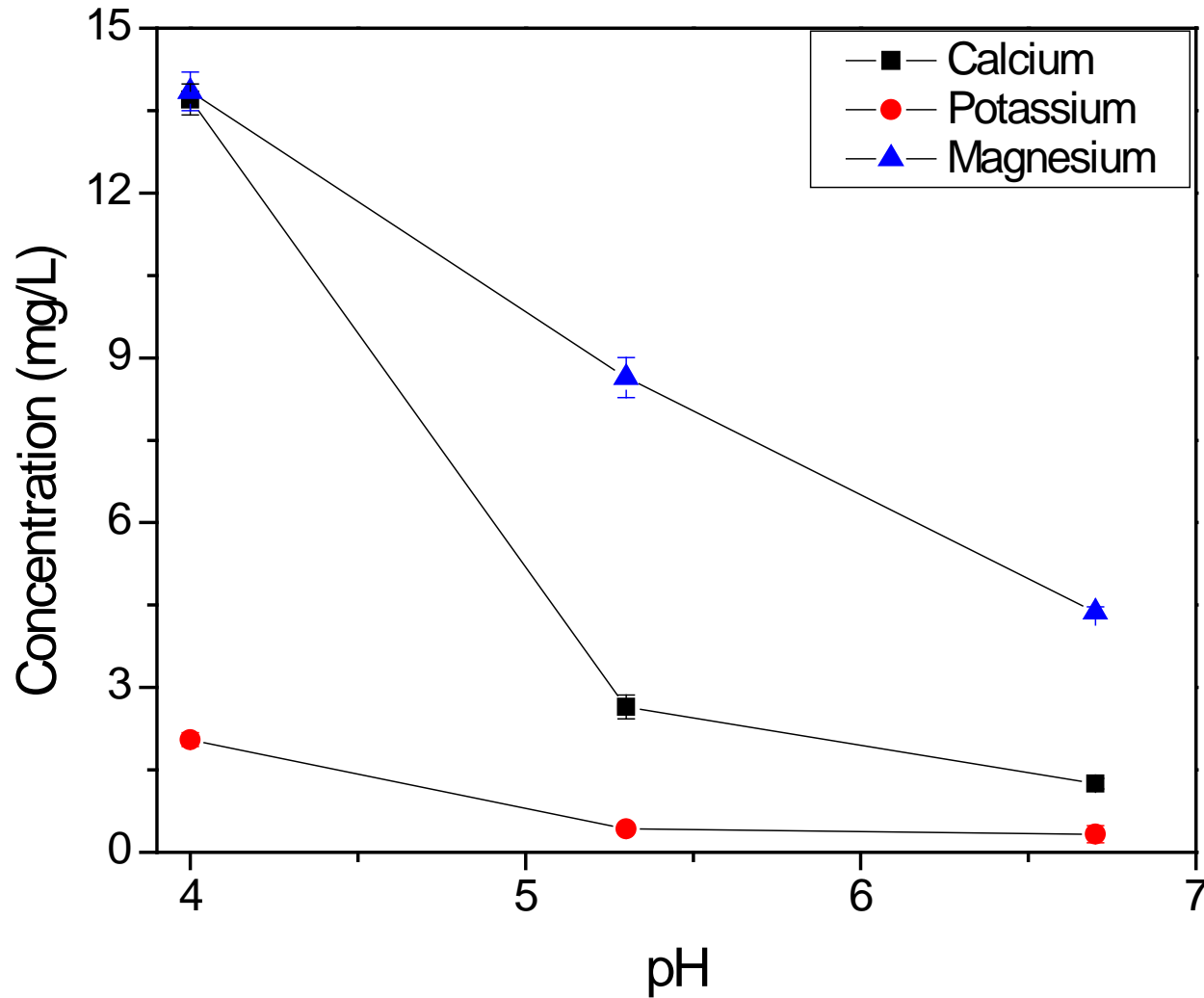


Blue: Unground  
Maroon: Ground



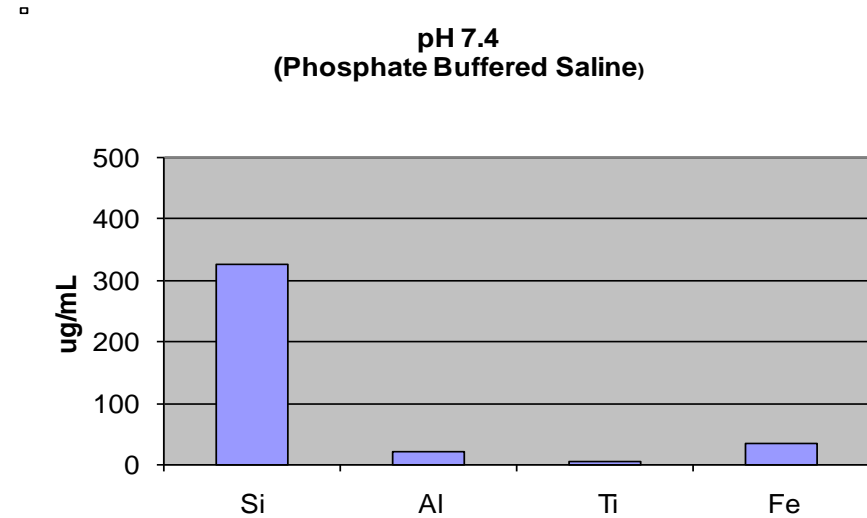
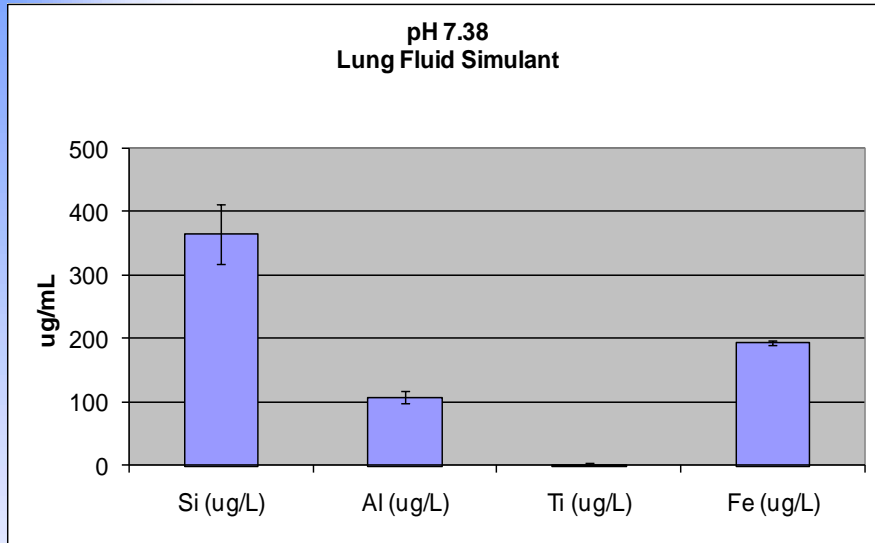


# Effect of pH on Leaching





# Lung Fluid Simulant



## Tyrode's solution modified to approximate Gamble's solution\*

1.8 mM  $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$ , 1.12 mM  $\text{MgCl}_2$ , 2.68 mM  $\text{KCl}$ , 11.9 mM  $\text{NaHCO}_3$ , 136.89 mM  $\text{NaCl}$ , 0.42 mM  $\text{NaH}_2\text{PO}_4$ , 5.55 mM D-Glucose, 10 mM  $\text{NH}_4\text{Cl}$ , 0.2 mM trisodium citrate, 6 mM glycine, 0.5 mM  $\text{Na}_2\text{SO}_4$



# Solubility Summary

---

- Grinding of lunar dust simulant leads to increased dissolution in buffers of different pH
- Decreases in pH lead to increased leaching from lunar simulant
- Use of lung fluid simulant does not lead to significant increase in leaching
- Where to go from here?
  - Different time points?
  - Lunar dust?
- Thanks to Mike Kuo for ICP-MS measurements

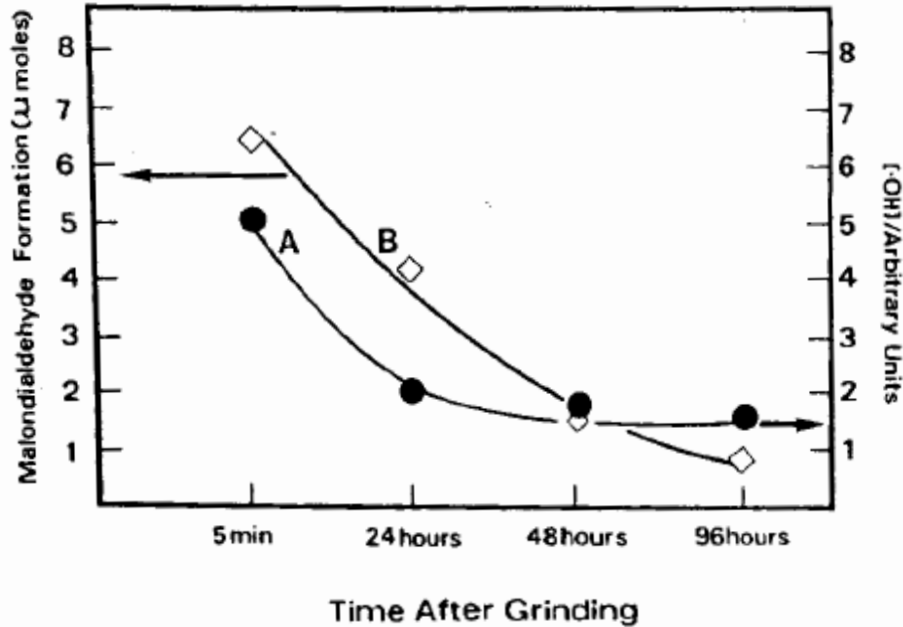


# Cell Culture

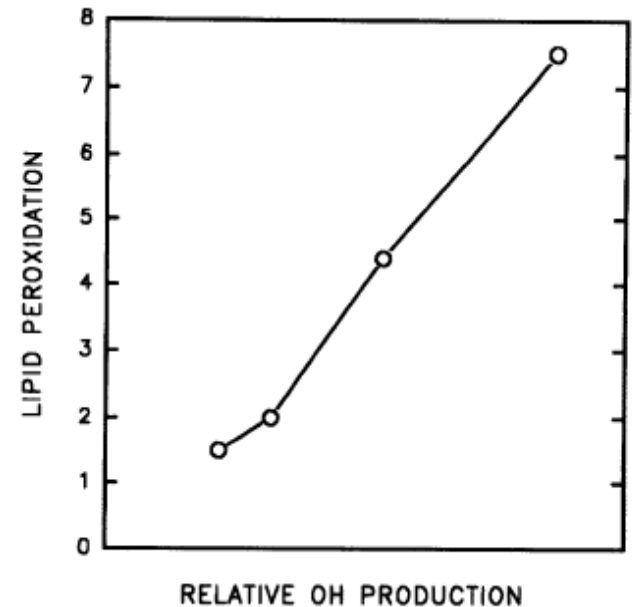




# Direct Toxicity of Quartz



Parameter	Aged Si	Fresh Si
Hemolysis <sup>b</sup>	2 ± 1%	39 ± 1%
Membrane leakiness <sup>c</sup>	15 ± 2%	58 ± 8%
Lipid peroxidation <sup>d</sup>	1.5 ± 0.4 μmol mal	7.5 ± 0.6 μmol mal



- Grinding of quartz also leads to direct toxicity *in vitro*
- Ability of ground silica to oxidize lipids is directly correlated to the number of radicals produced in solution and “freshness” of silica



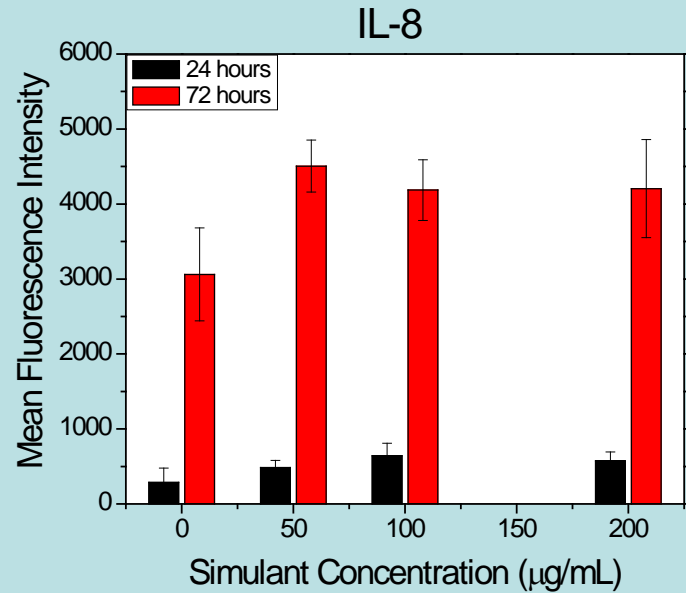
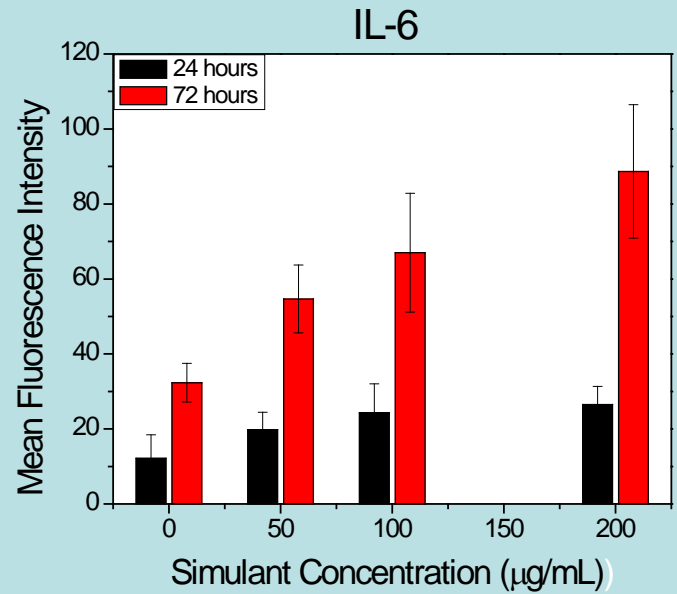
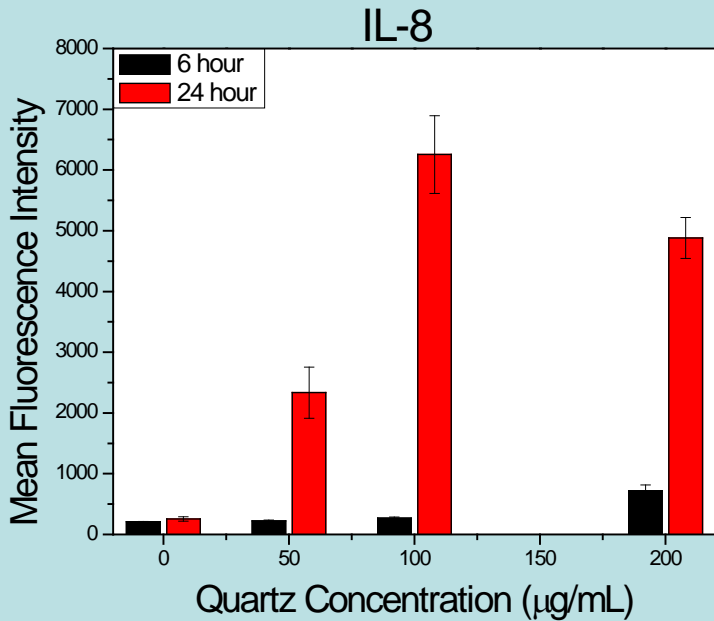
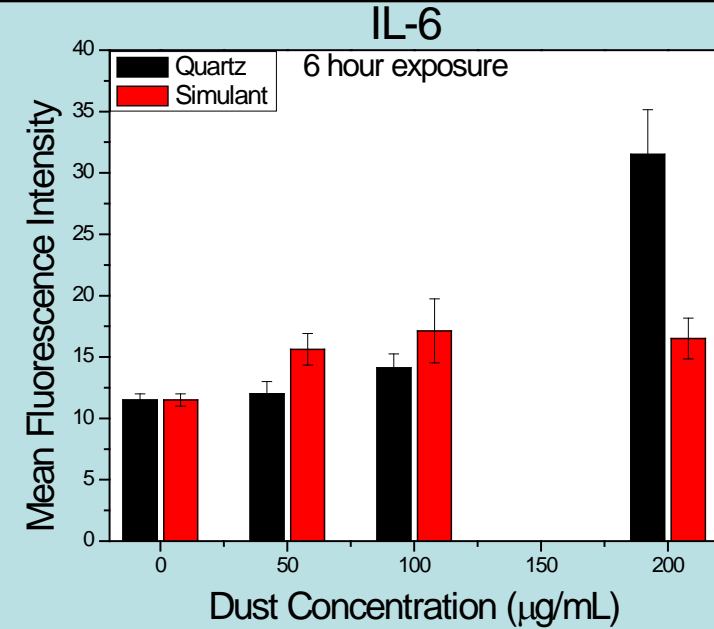
# Techniques

- A549 alveolar epithelial cells or BEAS-2B bronchial epithelial cells grown 72 hours
- Treatment media prepared by adding 10 mg of sample to 10 mL media w/o FBS (A549: F12K, BEAS-2B: GTSF-2)
  - Dilutions prepared (200, 100, and 50  $\mu\text{g/mL}$ ) from stock
  - Growth media removed from cells and 1 mL treatment media added
  - Cells incubated in treatment media for 6, 24, or 72 hours
- Media removed and centrifuged (5 min, 6000 rpm) to remove dust or cellular debris
- Supernatants tested for inflammatory mediators (IL-8, IL-6, and TNF- $\alpha$ )
- MTT toxicity testing also performed (mitochondrial activity)



# Cytokine Production

Unground JSC-1A-vf & Min-U-Sil 5

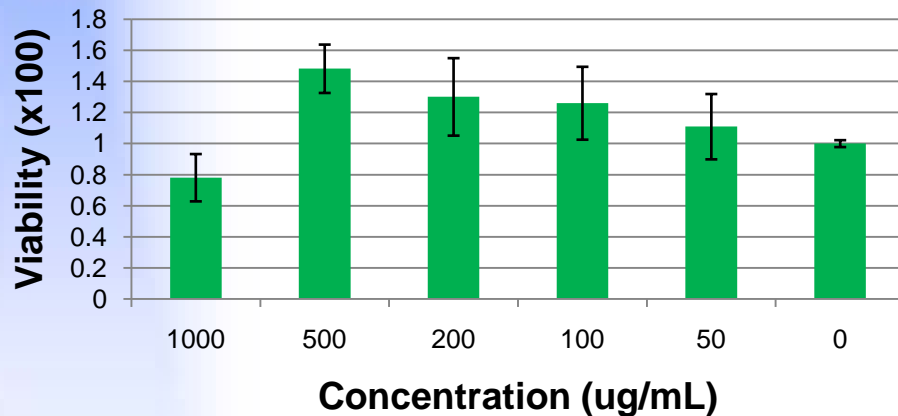


Ground JSC-1A-vf

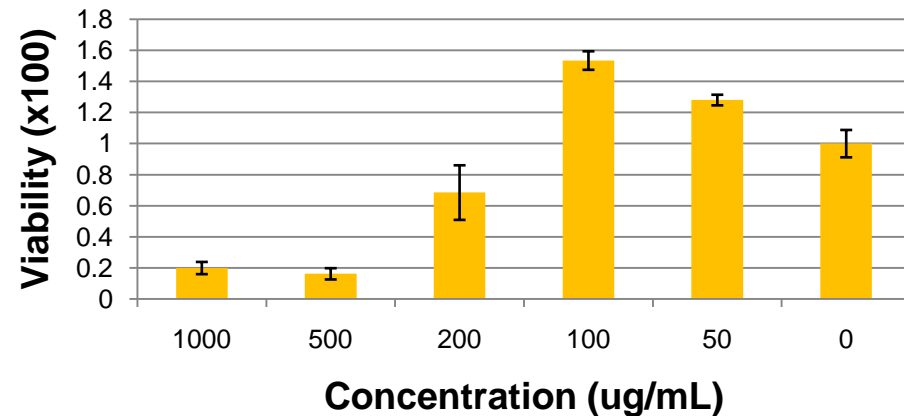


# A549 Viability

**JSC-1A-vf**  
Unground



**Min-U-Sil 5**  
Unground

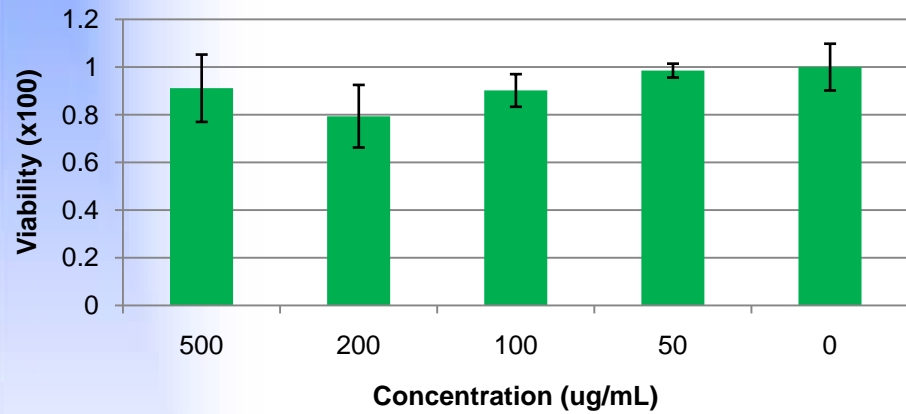


- Only highest concentration of simulant shows toxic effects; could be due to blockage of light by the dust
- Quartz is more toxic at lower concentrations

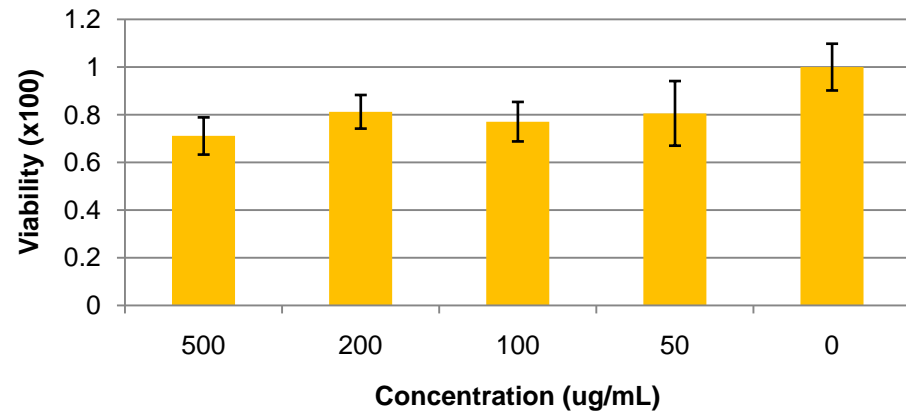


# A549 Viability

**JSC-1A-vf  
Ground**



**Min-U-Sil 15  
Ground**

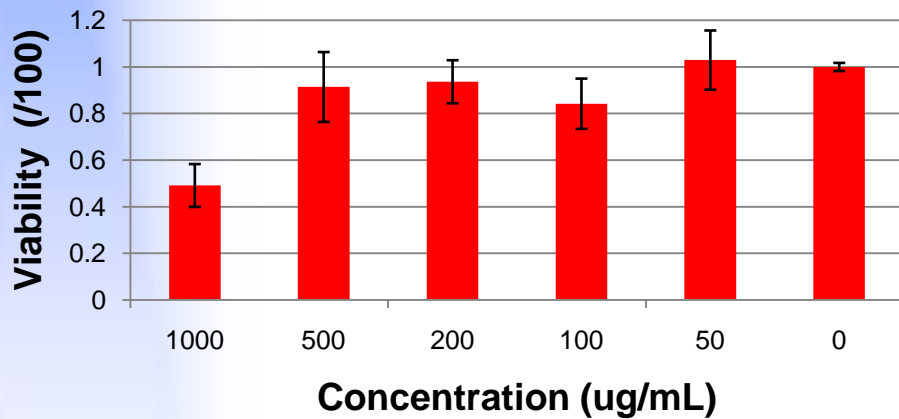




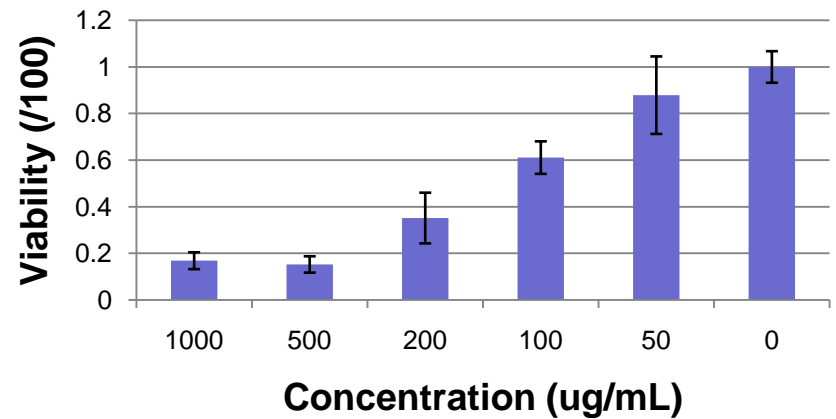


# BEAS-2B Viability

**JSC-1A-vf**  
Unground



**Min-U-Sil 5**  
Unground

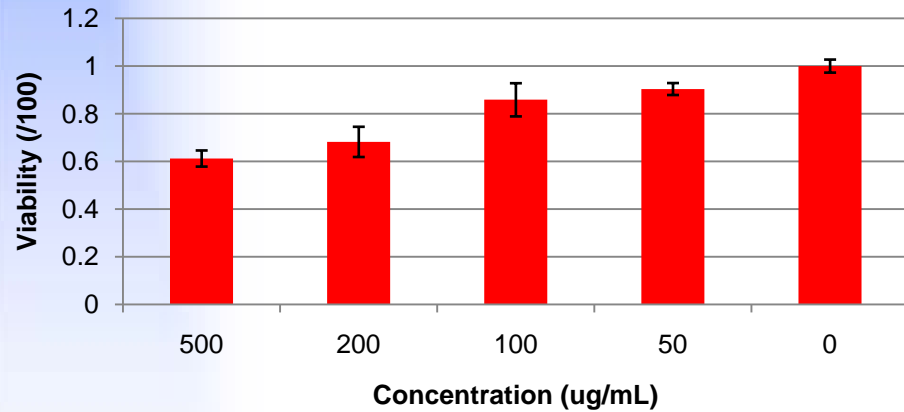


- Little toxicity seen for JSC-1A-vf
- Quartz shows relatively high toxicity even at low concentrations

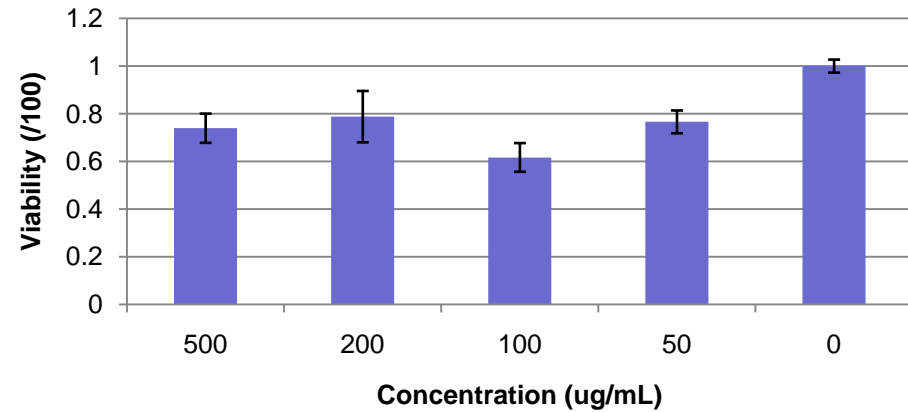


# BEAS-2B Viability

**JSC-1A-vf**  
Ground



**Min-U-Sil 15**  
Ground





# Cell Culture Conclusions

- Unground quartz causes an increase in IL-6 and IL-8 levels in A549 cells
  - Time dependence seen for IL-8 increase
- Unground JSC-1A-vf only causes noticeable IL-6 increase at highest concentration tested
- Ground JSC-1A-vf also produces IL-6 and IL-8 in A549
- Unground quartz is toxic to both A549 and BEAS-2B cells
  - Higher concentration required in A549
- Unground simulat only shows toxicity at highest concentration tested (1 mg/mL)



# Acknowledgements

Antony Jeevarajan

Dianne Hammond

Lunar Airborne Dust Toxicity  
Assessment Group (LADTAG)

Bo Chen

Maureen McCarthy

Mike Kuo

Kelley Bradley

Camil Liceaga

Kristyn Bales

