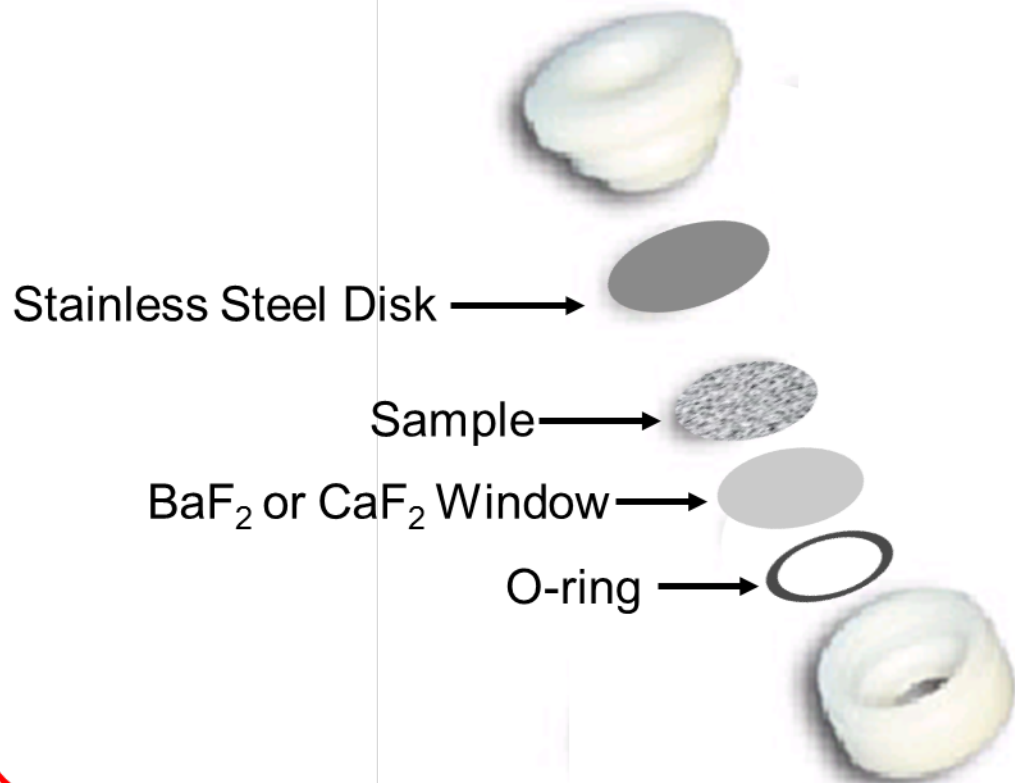


## Supplementary Material: Raman Examinations of A<sub>2</sub>TeC<sub>2</sub> Samples

- Raman spectra were recorded using a Renishaw inVia Raman Microprobe. Two lasers were used: 514 nm and 785 nm with similar results. Results with the 514 nm laser are reported herein.
- The sample ampules were opened in an argon box and loaded into the ambient barrier cell shown on slide 2.
- It was necessary to use a very low laser power level and to defocus (spread) the laser spot to avoid sample decomposition due to laser heating or perhaps photochemical reactions.
- The Raman results indicate that the Li<sub>2</sub>TeC<sub>2</sub> sample does not contain the acetylide anion.
- Crystalline and amorphous tellurium are detected to varying extents in both samples depending on the spot probed and the length of time the sample spent in the ambient barrier cell. Over time scales of several hours the samples turned from greenish yellow to light brown, indicating some decomposition.
- They also showed evidence of decomposition after opening of the ampule and several days of storage in a capped bottle kept in the glovebox.
- **The Raman results are consistent with the sodium compound being of the A<sub>2</sub>TeC<sub>2</sub> type but the lithium compound appears either to be some other phase or to have decomposed before (or during) Raman examination.**



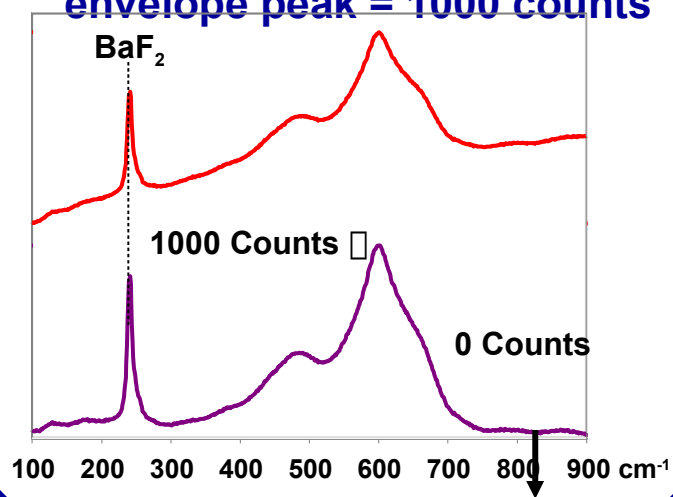
## Demountable Ambient Barrier Cell



**Raman Instrument: Renishaw inVia Raman Microscope at ANL's Center for Nanoscale Materials (785, 633, 514, 442, 325 nm excitation lasers)**

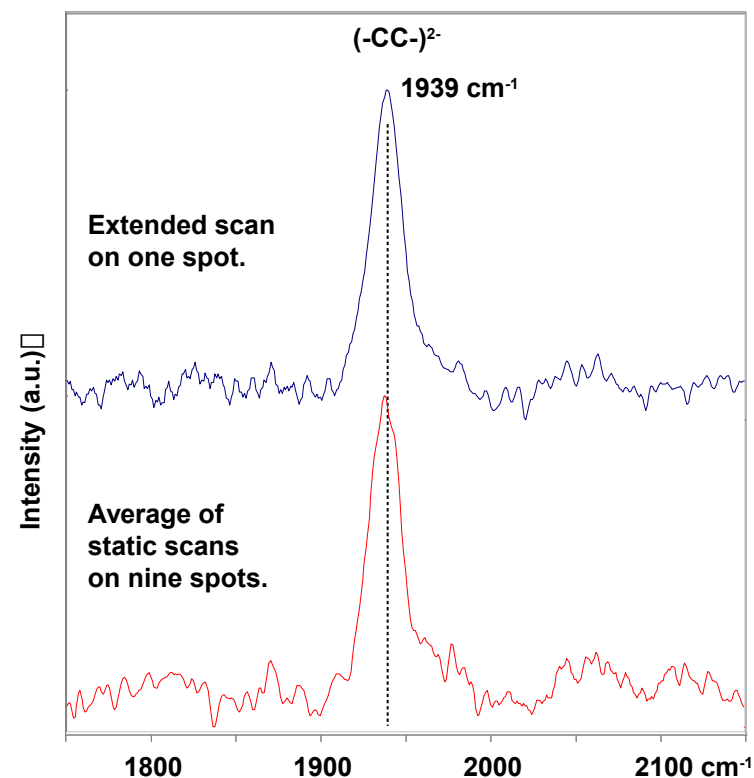
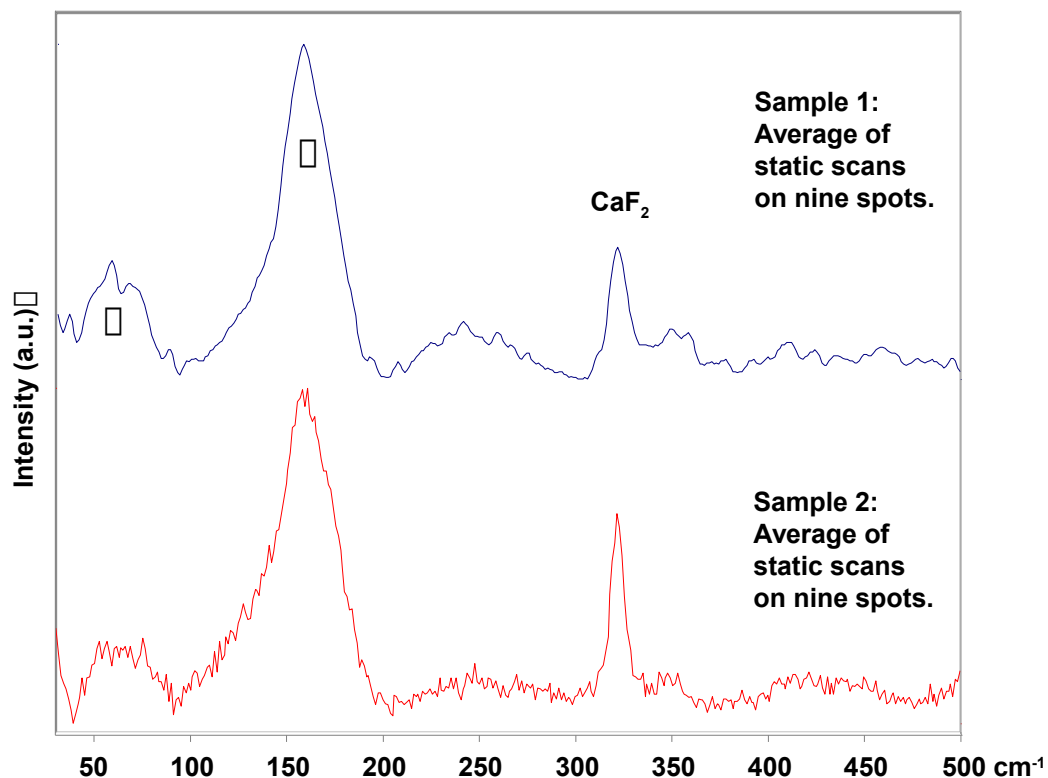
## Procedure

- Record spectra (at CNM)
- Extrapolate to a horizontal baseline
- Reset intensity range:  
baseline = 0 counts  
envelope peak = 1000 counts



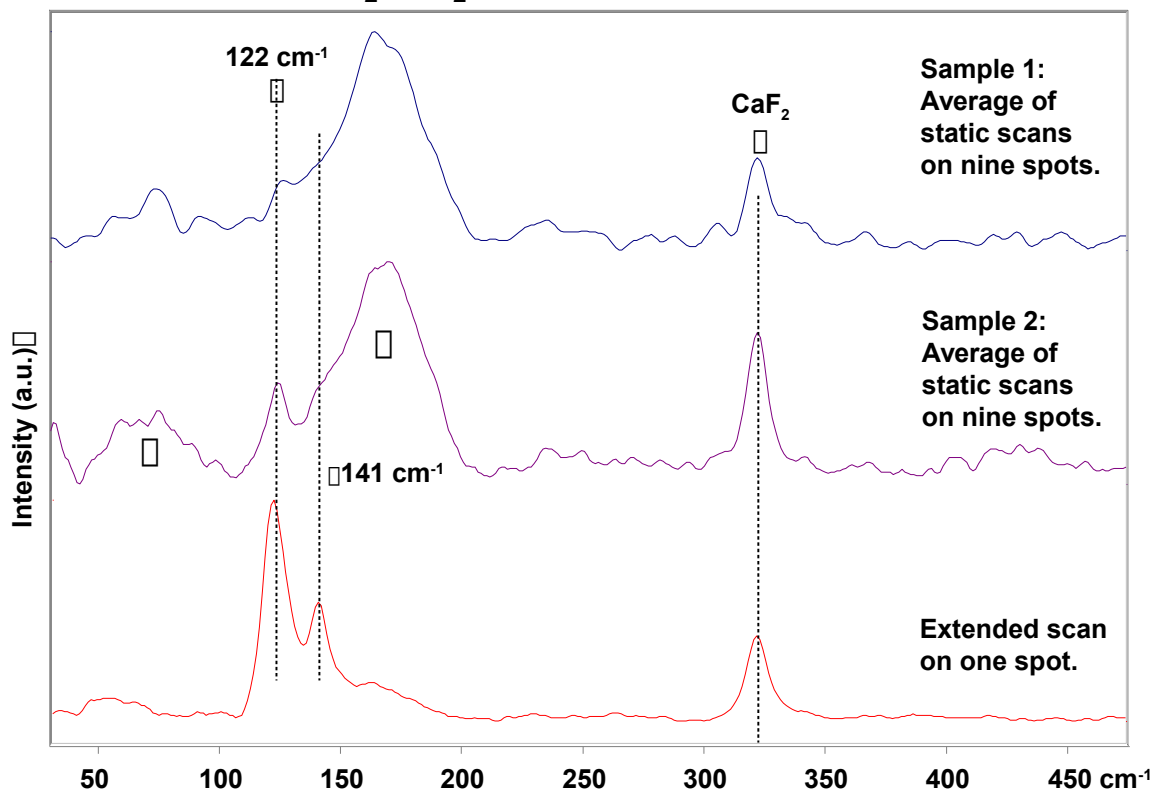
Two samples of  $\text{Na}_2\text{TeC}_2$  from the same ampule were investigated. A  $\text{CaF}_2$  window was used in the atmospheric barrier cell. Raman scattering was observed in two spectral regions shown below. The only band attributable to  $\text{Na}_2\text{TeC}_2$  is the acetylide stretching mode at  $1939\text{ cm}^{-1}$  seen in the right inset below. The bands marked with  $\square$  in the left inset are most probably due to amorphous tellurium as subsequent slides will indicate.

### $\text{Na}_2\text{TeC}_2$ : 514 nm excitation

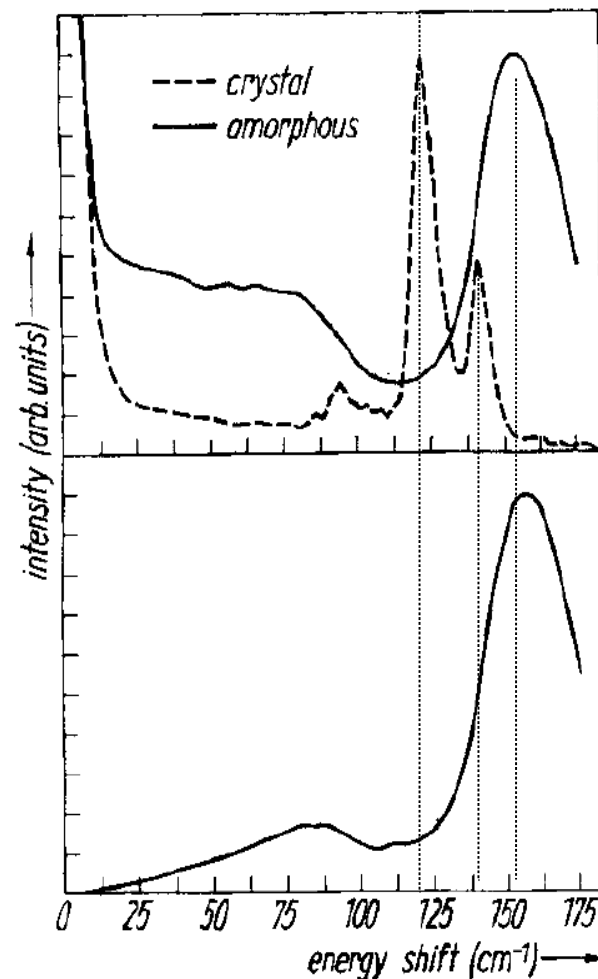


Two samples of “ $\text{Li}_2\text{TeC}_2$ ” (each from a different ampule) were investigated. A  $\text{CaF}_2$  window was used in the atmospheric barrier cell. No Raman scattering was observed in the acetylide spectral region. Several types of spectra were seen in the spectral region shown below, depending on location and time in the cell. The bands at 141 and 122  $\text{cm}^{-1}$  are undoubtedly due to crystalline Te. The bands marked with  $\square$  are most probably due to amorphous tellurium.

$\text{Li}_2\text{TeC}_2$ : 514 nm excitation



M. H. Brodsky, R. J. Gambino, J. E. Smith, Y. Yacoby, “The Raman Spectrum of Amorphous Tellurium,” *phys. stat. sol. (b)* **52**, 609 (1972).



Another old but still relevant reference.

A.S. Pine and G. Dresselhaus, "Raman Spectra and Lattice Dynamics of Tellurium," PHYSICAL REVIEW B 4, 356 (1971).

TABLE I. Raman-scattering data in tellurium.

| Mode                      | Temperature 295 °K            |                               | Temperature 90 °K             |                               |
|---------------------------|-------------------------------|-------------------------------|-------------------------------|-------------------------------|
|                           | Frequency (cm <sup>-1</sup> ) | Linewidth (cm <sup>-1</sup> ) | Frequency (cm <sup>-1</sup> ) | Linewidth (cm <sup>-1</sup> ) |
| $E_{TO}^{\pm}$            | 92.2 ± 0.5                    | 4.8 ± 0.5                     | 95.2 ± 0.5                    | 1.2 ± 0.5                     |
| $E_{LO} - E_{TO}$         | 12 ± 2                        | ...                           | ...                           | ...                           |
| $E_{TO}^{+} - E_{TO}^{-}$ | ...                           | ...                           | -0.60 ± 0.1                   | ...                           |
| $A_1$                     | 120.4 ± 0.5                   | 6.4 ± 0.5                     | 123.4 ± 0.5                   | 1.5 ± 0.5                     |
| $E_{TO}^{\pm}$            | 140.7 ± 0.5                   | 2.8 ± 0.5                     | 142.9 ± 0.5                   | 0.4 ± 0.4                     |
| $E_{LO} - E_{TO}$         | ...                           | ...                           | 0.6 ± 0.2                     | ...                           |
| $E_{TO}^{+} - E_{TO}^{-}$ | ...                           | ...                           | -0.26 ± 0.1                   | ...                           |

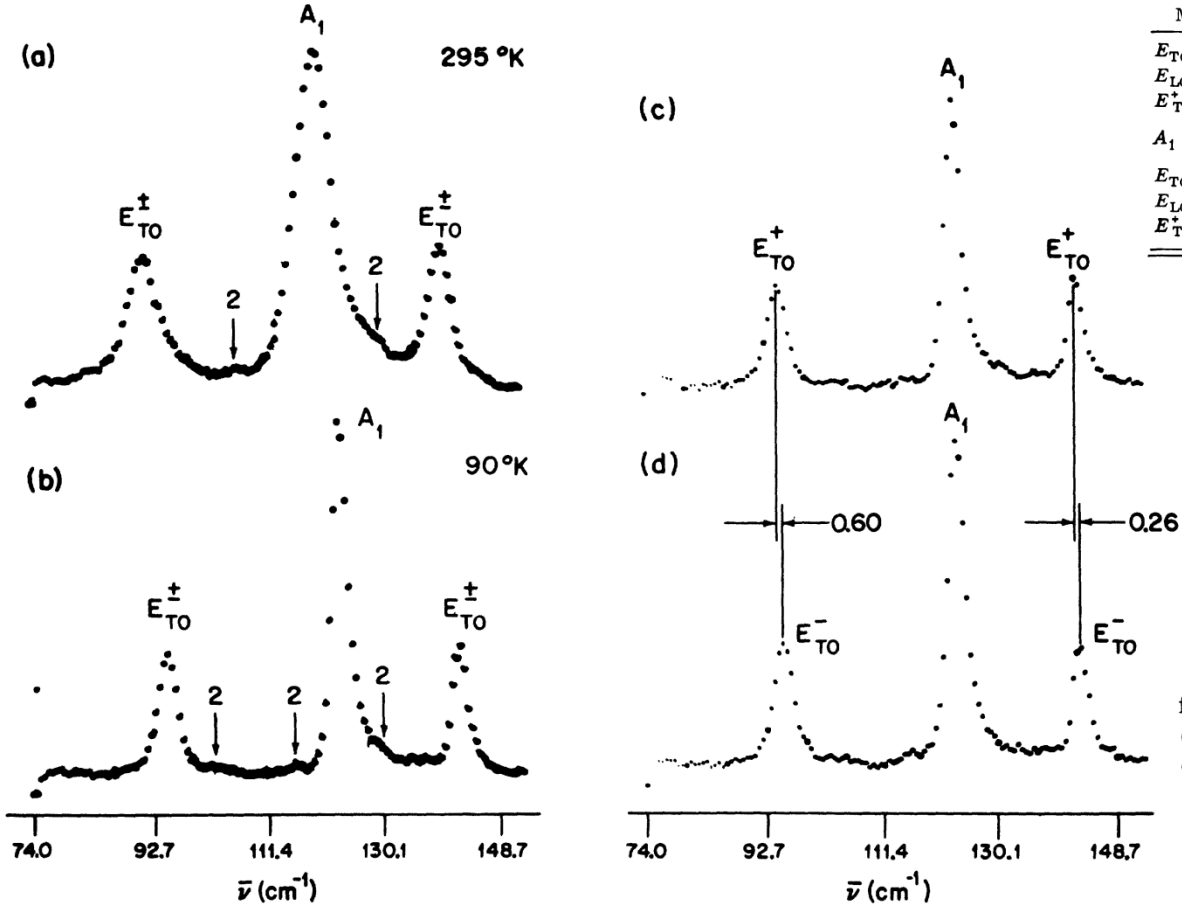


FIG. 1. Raman spectra from tellurium,  $\vec{q} \parallel c$  axis; (a)  $z(x, x+y)\bar{z}$ , 295 °K; (b)  $z(x, x+y)\bar{z}$ , 90 °K; (c)  $z(+, x+y)\bar{z}$ , 90 °K; (d)  $z(-, x+y)\bar{z}$ , 90 °K; (e) instrumental profile, 100- $\mu$  slits.

