

Deuterium Isotope Effect on the Crystal Structures of Pyrene: A Lab XRPD Study

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Laboratory XRPD data were collected from capillary samples of perdeuterated pyrene (C₁₆D₁₀) and protonated pyrene (C₁₆H₁₀) in the range $T = 80\text{--}140\text{K}$ [1]. Unit cell volumes were determined by Pawley refinement (*ca.* 120 reflections to 2 Å resolution), yielding:

(i) $V(T) = (0.1511 \pm 0.008672)T + (1001.9 \pm 1.0)$, $r^2 = 0.9743$, for C₁₆D₁₀ (form I);

(ii) $V(T) = (0.1449 \pm 0.006194)T + (1004.8 \pm 0.7)$, $r^2 = 0.9803$, for C₁₆H₁₀ (form I).

A comparison of slopes ($P = 0.553$) and elevations ($P < 0.0001$) within *Prism v4.02* confirmed that whilst the difference between the slopes is not significant, the perdeuterated crystal structure is significantly more compact than its hydrogenous counterpart at any given T . A likewise comparison of form II C₁₆D₁₀ with form II C₁₆H₁₀ produced the same conclusion.

The quality of the XRPD analysis was assessed against a neutron powder diffraction analysis of C₁₆D₁₀ [2]. The principal differences of note are the superior precision of the best-fit parameters, plus the superior goodness of fit, obtained with the neutron analysis, e.g.:

(i) $V(T) = (0.1496 \pm 0.001047)T + (1002.0 \pm 0.2)$, $r^2 = 0.9993$, for C₁₆D₁₀ (form I; HRPD, ISIS Facility, RAL).

[1] Xu X., *Ph.D. Thesis*, University of Strathclyde, Glasgow, UK, 2005. [2] Love S.W., *Ph.D. Thesis*, University of Strathclyde, Glasgow, UK, 1997.

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