THE PHYSICAL CONSTANTS OF p-NITROPHENYL α -D-GALACTO-PYRANOSIDE AND ITS TETRA-O-ACETYL DERIVATIVE*

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The physical constants of the above compounds were the subject of a "correction" by Helferich and Jung (1955).‡ However, the melting points these authors assign to them are still seriously in error.

The melting point of tetra-O-acetyl p-nitrophenyl α -D-galactopyranoside (129–130 °C, H. & J.) is 147 °C for material dried at 100 °C. Air-dried after recrystallization from ethanol, the tetra-acetate showed preliminary softening at 129–131 °C. Recrystallization from toluene-cyclohexane gave material with a first m.p. at 137 °C and a second at 147 °C; material from chloroform-diethyl ether had m.p.'s 96–100 and 147 °C. The α -tetra-acetate presumably forms molecular compounds with the solvents; similar behaviour is shown by the β -isomer with m.p.'s 128 and 146 °C for air-dried material that had been crystallized from ethanol.

The monohydrate of p-nitrophenyl α -D-galactopyranoside softens over the range 85 ± 5 °C (or melts and resolidifies in an apparatus preheated to 110–120 °C) and melts at 151–153 °C with immediate resolidification to re-melt at 173 °C (m.p.'s 83–85 and 146–149 °C, H. & J.). Material dried at 60 °C or prepared by evaporating a warm solution melts at 151–153 °C and again at 173 °C. Material dried at 100 °C has the single m.p. 173 °C (136–140 °C, H. & J.). The three m.p.'s are apparently those of the monohydrate, an intermediate hydrate (approximately galactoside $+0.5 \rm H_2O$; preparations were rather variable), and the anhydrous compound.

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[‡] Helferich, B., and Jung, K. N. (1955).—Liebigs Ann. 595: 242.