Preparation of o-nitrophenyl-b-D-galactopyranoside

after Seidman,M., and Link, K.P.

See Babers & Goebel, JBC 105:473, 1934.; Glaser & Wulwek, Bioch. Z. 145,514,'24

42 g. o-nitrophenol in sol. 16.8 g NaOH/420 ml H_2O. Add sol. 88 g acetobrom-galactose in 620 ml acetone. Keep at r.t. 5h. Remove solvent under reduced pressure-- filter off long needles, and concentrate further until crystals no longer come out. Wash ppt. water, air dry, recr. 95% EtOH. Weight: 56g. mp 172.5. (onpg-$Ac0_4$)

To deacetylate, lg. onpg-OAc_4 in 50 ml MeOH and 1 ml. .4N $Ba(OMe)_2$ added. Refrigerate, shake periodically. After 4 h. , clear sol., then crystals as long hairlike needles. After 24 h. conc. under red. pr. to 10 ml., and a quant. yield of onpg is obtained. Recr. 2x EtOH. MP 196-7.

For Acetobrom galactose:

Gal-OAc_5 (accdg. to USBS c440) , 50 g. with 50 ml HBr/HAcO (EK) Stand 2h. Add 200 cc $CHCl_3$, pour mix into 1 l. icewater, separate, wash $CHCl_3$ layer with $NaHCO_3$, then water (cold). Dry over Na_2SO_4. Evap. Take up syrup in Et_2O to crystallize. If necessary, ass Petether to turb. to initiate cryst.