enediamine dihydrochloride was found to be 0.86 c./m./mg. This indicates that 2.6% of the succinic acid radioactivity was in the methylene carbon atoms.

Oxidation of Malic Acid.—To a solution of 109.7 mg. of C^{14}-labeled malic acid (275 c./m./mg. or 30,100 c./m. total.) in 10 ml. of 1.0 *N* sulfuric acid in a 100-ml. flask equipped with nitrogen inlet bubbler, reflux condenser and dropping funnel was added 20 ml. of 0.15 *M* chromic acid solution during two hours on the steam-bath.

The carbon dioxide evolved was collected in carbonate-free sodium hydroxide solution and precipitated with barium chloride to give 326 mg. (101%) of barium carbonate. The specific activity was found to be 81.5 c./m./mg. or 26,600 c./m. total. Acetic acid was obtained from the residual solution upon steam distillation. It was converted to barium acetate (80% yield based on titer of steam distillate) which was recrystallized from water. From the specific activity (2.0 c./m./mg.) and the theoretical yield (112 mg.), the activity in the α and β carbon atoms of the malic acid, 224 c./m., can be determined accurately without interference from carboxyl activity. Incomplete recovery of the carbon dioxide and overoxidation of the malic acid is assumed to have caused the loss of carboxyl activity.

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Acetylation of D-Psicose

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In connection with our studies[2] on the chromatography of acetylated molasses fractions, it became of interest to investigate the behavior of the rare ketose D-psicose (synonyms D-allulose and D-erythrohexulose) under mild acetylating conditions since Zerban and Sattler[3] have shown that this ketose may be a constituent of some molasses fractions. Under conditions of mild acetylation the ketoses may form acetates of the cyclic or acyclic types.[4] In the work herein described D-psicose was acetylated at low temperature with zinc chloride and acetic anhydride and the resultant product was subjected to chromatographic analysis. Five fractions (Table I) were obtained and from the major fraction there was isolated the previously described[5] crystalline *keto*-D-psicose pentaacetate. The main product then of the direct acetylation of D-psicose under mild conditions is the acyclic acetate.

TABLE I

CHROMATOGRAM OF ACETYLATED D-PSICOSE

Zone	Approx. time, sec., for distinct zone formation	Zone position in mm. from top of column	Zone yield, g.
Top	60	0–3	0.017
1	180	21–27	.064
2	180	63–73	.095
3	30	115–122	.183
4	10	126–129	.037
		Total	.396 (84.6%)

Experimental

The D-psicose sirup obtained from the deacetylation of 500 mg. of *keto*-D-psicose pentaacetate[5] was treated with 10 ml. of acetic anhydride and 0.3 g. of freshly fused zinc chloride for twenty hours at 0–3°. The reaction mixture was then poured onto 40 g. of ice and water under good agitation and after thirty minutes of stirring the *p*H of the mixture was adjusted to 6 with sodium bicarbonate. The mixture was then extracted with three 25-ml. portions of chloroform. A sirup was obtained on solvent removal; yield 470 mg.

The above sirup (468 mg.), dissolved in 10 ml. of benzene, was added at the top of a 170 × 44 mm. (diam.) column of 100 g. of 5 parts Magnesol[6]/1 part Celite (by wt.) pre-wet with benzene. The chromatogram was developed with 600 ml. of 100 parts benzene/1 part ethanol (by vol.). Five zones were detected on the extruded column with the permanganate streak indicator (1% potassium permanganate in 2.5 *N* sodium hydroxide) and were eluted with acetone (Table I). Well-formed elongated prisms were obtained from a 95% ethanolic solution of zone 3 and were identified as *keto*-D-psicose pentaacetate; yield 90 mg., m. p. 64–65° (mixed melting point unchanged), $[\alpha]^{23}\mathrm{D}$ −21.0° (*c* 4, chloroform). Accepted values[5] for *keto*-D-psicose pentaacetate are: m. p. 63–65°, $[\alpha]^{29}\mathrm{D}$ −21.5° (*c* 3, chloroform). No crystalline products were obtained from the other zones.

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[1] Sugar Research Foundation Fellow of The Ohio State University Research Foundation (Project 190).

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Rearrangement of Alkyl Sulfites to Alkanesulfonate Esters

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Although a number of references on the formation of *salts* of alkanesulfonic acids from the reaction of alkyl sulfites with sodium or potassium hydroxides have appeared,[1,2,3,4] only one direct conversion of an alkyl sulfite to an *ester* of an alkanesulfonic acid has been described.[3] In that case, only a very small amount of methyl methanesulfonate resulted when dimethyl sulfite and excess methyl iodide were heated in a sealed tube for twelve hours. We have now found that *catalytic* amounts of tertiary amines, such as tri-*n*-butylamine, are excellent agents for promoting the rearrangement of dimethyl sulfite to methyl methanesulfonate, in yields as high as 56%. Dimethyl ether and sulfur dioxide were the other products. Diethyl sulfite, with the same amine, gave lower yields of either ethyl ethanesulfonate or

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