solution. The combined barium sulfate and compound VI was collected and extracted with hot water. Addition of sodium nitrate to the filtrate afforded the crude iodonium nitrate VI, decomposing at 233°; yield 77%. Recrystallization from water raised the decomposition temperature to 236°.

Anal. Calcd. for $C_{12}H_7O_6N_2I$: I, 32.79. Found: I, 32.91, 32.78.

VI was characterized by reducing it to the known 2-iodo-4′-nitrobiphenyl. A suspension of VI in alcohol was treated with just *enough* sodium to bring about complete solution. Addition of water afforded 2-iodo-4′-nitrobiphenyl, m. p. 98–99°, which when mixed with an authentic sample showed no depression of the melting point.

VI was converted into the iodonium chloride (II) by treating its aqueous solution with dilute hydrochloric acid. The chloride was very insoluble in water and alcohol. It decomposed at 279–282°; yield almost quantitative.

Anal. Calcd. for $C_{12}H_7O_2NICl$: Cl and I, 45.16. Found: Cl and I, 45.28, 45.16.

The dinitration[7] of IV afforded 4,4′-dinitrodiphenyleneiodonium sulfate. It was very insoluble in water and alcohol. For purification it was extracted repeatedly with hot alcohol. The yield was 95%; decomposition temperature 281–283°.

Anal. Calcd. for $C_{24}H_{12}O_{12}N_4SI_2$: I, 30.35; S, 3.83. Found: I, 30.75, 30.66; S, 3.89, 3.79.

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[7] Sandin, McClure and Irwin, THIS JOURNAL, **61**, 3061 (1939).

DEPARTMENT OF CHEMISTRY
UNIVERSITY OF ALBERTA
EDMONTON, CANADA

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NEW COMPOUNDS

Tetrabenzoyl-α-D-glucopyranosyl Bromide–Carbon Tetrachloride Addition Compound[1]

Ten grams of pentabenzoyl-α-D-glucopyranose was dissolved in 50 ml. of hot glacial acetic acid and cooled to room temperature. To this was added 40 ml. of hydrobromic acid–acetic acid solution (30–32% hydrobromic acid). After two hours at room temperature the clear yellow solution was poured into 1 liter of ice-water and the product separated by filtration. This solid was dissolved in 500 ml. of carbon tetrachloride and the solution washed once with distilled water, twice with saturated sodium bicarbonate solution, once more with water, filtered through glass wool, and dried over calcium chloride. After filtration, the product crystallized on addition of low boiling petroleum ether to the carbon tetrachloride solution. Three recrystallizations from carbon tetrachloride and petroleum ether mixtures yielded colorless needles; m. p. 103–105° (uncor.), $[\alpha]^{22}D + 120°$ (*c*, 2.26, toluene), $[\alpha]^{25}D + 99°$ (*c*, 3.95, chloroform).

Anal. Calcd. for $C_{34}H_{27}O_9Br{\cdot}CCl_4$: C, 51.68; H, 3.35; halogen, 39.83. Found: C, 51.83; H, 3.49; halogen, 38.59 (gravimetric).

Recrystallization of the addition compound from ether and petroleum ether mixtures yielded the known tetrabenzoyl-α-D-glucopyranosyl bromide without the molecule of carbon tetrachloride of crystallization; m. p. 127–128°, $[\alpha]^{25}D + 146°$ (*c*, 2.05, toluene); + 119° (*c*, 3.62, chloroform).

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[1] Contribution from the Southern Regional Research Laboratory, one of the laboratories of the Bureau of Agricultural and Industrial Chemistry, Agricultural Research Administration, U. S. Department of Agriculture. Not copyrighted.

2-Chloromethyl-5-hydroxy-6-chloro-4-pyrone

2-Chloromethyl-5-hydroxy-4-pyrone (2-chlorokojic acid) was prepared from kojic acid[1] essentially as described by Kipnis, *et al.*[2]

Seven grams of 2-chlorokojic acid was shaken with 0.5 g. of anhydrous aluminum chloride in a 250-ml. flask and fitted with a reflux condenser (all-glass standard-taper connections). The mixture was then treated with 11.4 g. of sulfuryl chloride introduced through the top of the condenser. When the reaction had subsided the mass was carefully heated at 70° by a hemispherical mantle, and maintained at that temperature for about twenty minutes. The resulting material was a yellow solid which was then mixed with 50 ml. of water and filtered.

The substance was recrystallized from alcohol and then from chloroform. Finally, because of the reoccurrence of a faint red coloration, the crystals were washed with distilled water and then air-dried.

The compound consisted of white needles which became yellow upon storage. Yield 4.4 g. (51%) m. p. 139–141°. *Anal.*[3] Calcd for $C_6H_4Cl_2O_3$: Cl, 36.33. Found: Cl, 36.30.

The compound when treated with a dilute solution of ferric chloride gave a red coloration. The substance is assigned the probable structure of 2-chloromethyl-5-hydroxy-6-chloro-4-pyrone since it has been shown[4] that several 4-pyrones are most vulnerable to attack by a halogen at position 6.

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[1] The kojic acid used in this experiment was furnished by the Northern Regional Laboratory of the United States Department of Agriculture, Peoria, Ill.

[2] Kipnis, Soloway and Ornfelt, THIS JOURNAL, **70**, 4264 (1948).

[3] Analysis by Dr. Carl Tiedcke, Teaneck, New Jersey.

[4] See references to the formation of 2-hydroxymethyl-5-hydroxy-6-bromo-γ-pyrone and 2-methyl-5-hydroxy-6-bromo-γ-pyrone by Barham and Smits, *Transactions of Kansas Academy of Science*, **37**, 91–113 (1934).