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## Preparation of a sodium salt of 3-phosphoryl-D-glyceric acid

Soluble salts of 2-phosphoryl-D-glyceric acid and 3-phosphoryl-D-glyceric acid, free of heavy metal ions, were needed in studies of phosphoglycerate mutase now in progress in this laboratory. The synthesis of a sodium salt of 2-phosphoryl-p-glyceric acid has been described by Ballou<sup>1</sup>. A sodium salt of 3-phosphoryl-p-glyceric acid may be prepared from the purified barium salt by the following procedure.

Procedure

3-Phosphoryl-D-glyceric acid (3-PGA) was isolated as the barium salt from a yeast fermentation mixture<sup>2</sup>. The barium salt was recrystallized three times before conversion to the sodium salt\*.

9.5 g of Ba<sub>3</sub>PGA·<sub>2</sub>H<sub>2</sub>O was shaken with 200 ml of a 1:1 slurry of Dowex 50 (Na<sup>+</sup>) for 2 hours. The mixture was filtered and the resin was washed twice with 25 ml portions of water. Total volume of filtrate and washings was 150 ml. The solution, at pH 7, was concentrated in vacuo to 30-40 ml and filtered if not clear. Absolute methanol was added to 100 volumes and then n-hexane was added to appearance of the white salt and/or appearance of a second phase. The mixture was stored at room temperature to complete precipitation of the sodium salt. The salt was removed by centrifugation and washed with acetone. After drying in a vacuum oven at 55°, the stable white powder amounted to 4.5 g. Yield = 75% as Na<sub>2</sub>3PGA. The compound lost no weight on drying over P<sub>2</sub>O<sub>5</sub> at 78° for eight hours.

| Element analysis | Calculated<br>for C <sub>3</sub> H <sub>5</sub> O <sub>7</sub> PNa <sub>2</sub> | Found              |
|------------------|---|--------------------|
| % C              | 15.68   | 15.48**<br>13.10** |
| % P              | 13.48   | 13.10**            |
| % Na             | 20.00   | 19.87              |

 $[a]_{20}^{D}=-735^{\circ}\pm8^{\circ}$  (—0.82°, I d.cm tube, c= 1.11 mg as the free acid) in 1/3 volume of 25% (w/v) (NH<sub>4</sub>)<sub>6</sub>Mo<sub>7</sub>O<sub>24</sub>·<sub>4</sub>H<sub>2</sub>O\*\*\*\*.

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<sup>\*</sup> Mr. Lewis Pizer, a graduate student of this department, has prepared the sodium salt from the barium salt of phosphoglyceric acid as supplied by Schwarz Laboratories Inc., New York, without further purification. Yield = 37%.

Minimum value.

<sup>\*\*\*</sup> The high rotation of 3-PGA in molybdate solution is well known3. Customarily such measurements have been made in 1/3 volume of 25% (NH<sub>4</sub>)<sub>6</sub>Mo<sub>7</sub>O<sub>24</sub>·4H<sub>2</sub>O (8% molybdate in the final mixture) at neutral pH. The writer<sup>2</sup> has observed, however, that neither concentration of molybdate nor pH are critical. The same rotation was observed in mixtures containing from 2.5 to 12.5% molybdate and at pH values from 7 to 4. The rotation is lowered in alkaline solutions.