1. Procedure

In a 1-l. round-bottomed flask equipped with a reflux condenser are placed 157.5 g. (1 mole) of \( p \)-chloronitrobenzene, 160 g. (1 mole) of potassium xanthate (Note 1), and 450 ml. of 95% ethanol. This reaction mixture is heated under reflux on a steam bath for 48 hours. The crystalline product, which deposits from solution during the course of the reaction, is collected by filtration, crushed into small particles in a mortar, and washed twice with hot ethanol and once with hot water. The yield of \( p \)-nitrophenyl sulfide melting at 158–160° is 105–113 g. (76–82%). This product is pure enough for most purposes. Recrystallization from glacial acetic acid (15 ml. per gram) raises the melting point to 160–161°.

2. Notes

1. Potassium xanthate may be prepared in the following manner: With heating, 300 g. (5.36 moles) of potassium hydroxide is dissolved in 3 l. of absolute ethanol. The solution is then cooled in an ice bath, and the temperature is kept below 10° while carbon disulfide is added in portions with stirring until the solution is no longer alkaline; about 456 g. (360 ml., 5.95 moles) of carbon disulfide is required. The potassium xanthate is collected by suction filtration and air-dried on large sheets of filter paper; yield, 429–472 g. (50–55%).

3. Discussion

\( p \)-Nitrophenyl sulfide has been prepared by the reaction between \( p \)-chloronitrobenzene and sodium sulfide.\(^1\) This is not a practical means of preparation, however, because of the variety of substances formed.\(^2\) The method described has been published.\(^3\) A preparation of pure \( p \)-nitrophenyl sulfide has been reported from \( p \)-chloronitrobenzene and sodium thiosulfate.\(^4\)

References and Notes

ethanol (64-17-5)

acetic acid (64-19-7)

sodium thiosulfate (7772-98-7)

potassium hydroxide (1310-58-3)

carbon disulfide (75-15-0)

sodium sulfide (1313-82-2)

p-chloronitrobenzene (100-00-5)

potassium xanthate

p-Nitrophenyl sulfide,
Sulfide, bis-(p-nitrophenyl) (1223-31-0)