Phosphorus and Selenium NMR Spectroscopy

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Practical:

Abstract

Like most phosphines, PP3 is oxidised by reaction with chalcogens to form pentavalent phosphorus compounds.

You will be able to monitor the extent and regioselectivity of the reaction of PP3 with S and Se through the use of 31P (100%abundance) NMR spectroscopy after the addition of 1,2,3 and 4 equivalents of the oxidant.

Intended academic level

Duration

Outcomes

Materials

FT-NMR spectrometer with multinuclear capabilities. We have run this experiment using a JEOL JNM GX270 NMR spectrometer, although lower-field instruments should also suffice.1

Two NMR tubes for each group of students.

Two 10 mL volumetric flasks for each group of students.

Two small weighing boats for each group of students.

10 g tris(2-diphenylphosphinoethyl)phosphine. Typically, less than 1 2 g is used by each group of students. This reagent is also known as “tetraphos” or “PP3”; it can be purchased from several chemical companies as “tetraphos 2”. We have purchased our supplies from Strem Chemicals

10 g sulfur. Typically, less than 1 2 g is used by each group of students.

10 g selenium. Typically, less than 1 2 g is used by each group of students.

500 mL dichloromethane. Typically, only 100 mL is used by each group of students.

Costs

Further comments

Reading

Contact details

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