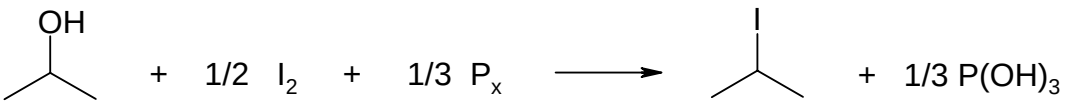
# 4025 Synthesis of 2-iodopropane from 2-propanol




| $C_3H_8O$ | | | $C_3H_7I$ | |
|---|---|---|---|---|
| (60.1) | (253.8) | (31.0) | (170.0) | (82.0) |

## Classification

**Reaction types and substance classes**
nucleophilic substitution
iodoalkane, alcohol

**Work methods**
heating under reflux, stirring with magnetic stir bar, shaking out, extracting, filtering, draining of gases, working with wash bottles, distilling with fractionating column, heating with oil bath

## Instruction (batch scale 10 mmol)

**Equipment**
10 mL round bottom flask, reflux condenser, 3 wash bottles, heatable magnetic stirrer, magnetic stir bar, separating funnel, distillation bridge, 10 cm Vigreux column, oil bath

**Substances**

| | |
|---|---|
| 2-propanol (dry) (bp 82 °C) | 0.601 g (0.765 mL, 10.0 mmol) |
| phosphorus, red | 0.124 g (4.00 mmol) |
| iodine | 1.29 g (5.08 mmol) |
| aqueous NaOH solution (0.5 M) | |
| molecular sieve 3Å | |
| diluted aqueous $NaHCO_3$ solution | |
| sodium sulfate for drying | |

**Reaction**
The procedure must be performed in a hood. The apparatus consists of a 10 mL round bottom flask with a magnetic stir bar and a reflux condenser. The reflux condenser is connected to three wash bottles one after the other. The first one, directly connected to the reflux condenser, is filled with molecular sieve, the second one is empty, the third one contains a 0,05 M aqueous sodium hydroxide solution.

1.29 g (5.08 mmol) iodine, 0.124 g (4.00 mmol) red phosphorus and 0.601 g (0.765 mL, 10.0 mmol) dry 2-propanol is added to the reaction flask. With stirring the reaction mixture is heated to boiling with an oil bath and refluxed for two hours.

**Work up**

After the reaction the reflux condenser is replaced by a distillation bridge and the crude product is directly distilled from the reaction mixture at a boiling temperature of 80-90 °C. The distilled product is transferred into a separating funnel and washed with diluted aqueous $NaHCO_3$-solution. The organic phase is separated and dried over sodium sulfate. After filtration the crude product is distilled through a 10 cm Vigreux column.

Yield : 1.21 g (7.12 mmol, 71%); bp 88 °C, colourless liquid; $n_D^{20}$ = 1.495

**Comments**

The use of molecular sieve as drying agent is recommended, otherwise the yield will be lower.

If there are small amounts of non reacted 2-propanol in the crude product it is no longer detectable after the washing procedure.

**Waste management**

**Recycling**

Molecular sieve can be regenerated.

**Waste disposal**

| Waste | Disposal |
|---|---|
| aqueous phase from extraction | solvent water mixtures, containing halogen |
| residue from the first distillation | dissolve in water, neutralize with diluted NaOH, then: solvent water mixtures, containing halogen |
| residue from the second distillation | organic solvents, containing halogen |
| sodium sulfate | solid waste, free from mercury |
| contents of the third wash bottle | domestic waste water |

**Time**

4 hours

**Break**

Before the first distillation and between the first and second distillation

**Degree of difficulty**

Easy

## Instruction (batch scale 100 mmol)

### Equipment

50 mL two-neck round bottom flask, Thielepape apparatus, reflux condenser, 3 wash bottles, heatable magnetic stirrer, magnetic stir bar, separating funnel, distillation bridge, 10 cm Vigreux column, oil bath

### Substances

| | |
|---|---|
| 2-propanol (dry) (bp 82 °C) | 6.01 g (7.65 mL, 100 mmol) |
| phosphorus, red | 1.24 g (40.0 mmol) |
| iodine | 12.9 g (50.8 mmol) |
| aqueous NaOH solution (0.5 M) | |
| molecular sieve 3Å | |
| diluted aqueous $NaHCO_3$ solution | |
| sodium sulfate for drying | |

### Reaction

The reaction must be performed in a hood. The apparatus consists of a 50 mL two-neck round bottom flask with a magnetic stir bar and on top a Thielepape apparatus combined with a reflux condenser. The reflux condenser is connected to three wash bottles one after the other. The first one, directly connected to the reflux condenser, is filled with molecular sieve, the second one is empty, the third one contains a 0,05 M sodium hydroxide solution. The Thielepape apparatus has a bottom frit with porosity P-0 or an extraction cone from glass with a bottom frit with porosity P-0. As an alternative the Thielepape apparatus may have a refluxing device, which is covered with glass wool.

12.9 g (50.8 mmol) of iodine is transferred into the Thielepape apparatus. 1.24 g (40.0 mmol) red phosphorus and 6.01 g (7.65 mL, 100 mmol) dry 2-Propanol is added into the reaction flask and with stirring the mixture is heated to boiling with the oil bath. By refluxing of solvent iodine in the reflux condenser is dissolved and transferred continuously into the reaction mixture. The oil bath can be removed after a short time, because the release of heat during the exothermic reaction results in refluxing without additional heating. When the reaction is finshed the mixture is refluxed for additional 30 minutes.

### Work up

At this stage the reflux of theThielepape apparatus is closed and the crude product is directly distilled into the Thielepape apparatus. The distilled product is taken from the apparatus and tranferred into a separating funnel. It is washed with diluted aqueous $NaHCO_3$-solution. The organic phase is separated and dried over sodium sulfate. After filtration of the drying agentum the crude product is distilled through a 10 cm Vigreux column.

Yield: 14.1 g (82.9 mmol, 83%,); bp 88°C, colourless liquid; $n_D^{20}$ = 1.495

### Comments

The use of molecular sieve as drying agent is recommended, otherwise yields are lower. The transference of solvent through the Thielepape apparatus should occur fast.

If there are small amounts of non reacted 2-propanol in the crude product it is no longer detectable after the washing procedure.

**Waste management**

**Recycling**
Molekular sieve can be regenerated.

**Waste disposal**

| Waste | Disposal |
|---|---|
| aqueous phase from extraction | solvent water mixtures, containing halogen |
| residue from the first distillation | dissolve in water, neutralize with diluted NaOH, then: solvent water mixtures, containing halogen |
| residue from the second distillation | organic solvents, containing halogen |
| sodium sulfate | solid waste, free from mercury |
| contents of the third wash bottle | domestic waste water |

**Time**
4 hours

**Break**
Before the first distillation and between the first and second distillation

**Degree of difficulty**
Easy

## Instruction (batch scale 1 mol)

**Equipment**
500 mL two-neck round bottom flask, Thielepape apparatus, reflux condenser, 3 wash bottles, heatable magnetic stirrer, magnetic stir bar, separating funnel, distillation bridge, 10 cm Vigreux column, oil bath

**Substances**

| | |
|---|---|
| 2-propanol (dry) (bp 82 °C) | 60.1 g (76.5 mL, 1.00 mol) |
| phosphorus , red | 12.4 g (0.400 mol) |
| iodine | 129 g (0.508 mol) |
| aqueous NaOH solution (0.5 M) | |
| molecular sieve 3Å | |
| diluted aqueous $NaHCO_3$ solution | |
| sodium sulfate for drying | |

**Reaction**
The procedure must be performed in a hood. The apparatus consists of a 500 mL two-neck round bottom flask and on top a Thielepape apparatus combined with a reflux condenser. The

reflux condenser is connected to three wash bottles one after the other. The first one, directly connected to the reflux condenser, is filled with molecular sieve, the second one is empty, the third one contains a 0.05 M sodium hydroxide solution. The Thielepape apparatus has a bottom frit with porosity P-0 or an extraction cone from glass with a bottom frit with porosity P-0. As an alternative the Thielepape apparatus may have a refluxing device, which is covered with glass wool.

29 g (0.508 mol) of iodine is added into the Thielepape-apparatus. 12.4 g (0.400 mol) red phosphorus and 60.1 g (76.5 mL, 1.00 mol) dry 2-propanol is added into the reaction flask. Then with stirring the mixture is heated to boiling with an oil bath. By the refluxing of the solvent iodine is dissolved and transferred continuously into the reaction flask. The oil bath can be removed after a short time, because the release of heat of the exothermic reaction results in refluxing without additional heating. When the reaction is finished the mixture is refluxed for additional 30 minutes.

**Work up**

At this stage the reflux of the Thielepape apparatus is closed and the crude product is directly distilled from the reaction mixture into the Thielepape apparatus. The distilled product is taken from the apparatus and transferred into a separating funnel. It is washed with diluted aqueous $NaHCO_3$ solution and dried over sodium sulfate. After filtration of the drying agent the crude product is distilled through a 10 cm Vigreux column.

Yield: 144 g (853 mmol, 85%); bp 88°C, colourless liquid; $n_D^{20}$ = 1.495

**Comments**

The use of molecular sieve as drying agent is recommended, otherwise yields will be lower. It is important, that the transference of solvent through theThielepape apparatus should occur fast.

If there are small amounts of non reacted 2-propanol in the crude product it is no longer detectable after the washing procedure.

**Waste management**

**Recycling**

Molekular sieve can be regenerated.

**Waste disposal**

| Waste | Disposal |
|---|---|
| aqueous phase from extraction | solvent water mixtures, containing halogen |
| residue from the first distillation | dissolve in water, neutralize with diluted NaOH, then: solvent water mixtures, containing halogen |
| residue from the second distillation | organic solvents, containing halogen |
| sodium sulfate | solid waste, free from mercury |
| contents of the third wash bottle | domestic waste water |

**Time**
5 hours

**Break**
Before the first distillation and between the first and second distillation

**Degree of difficulty**
Easy

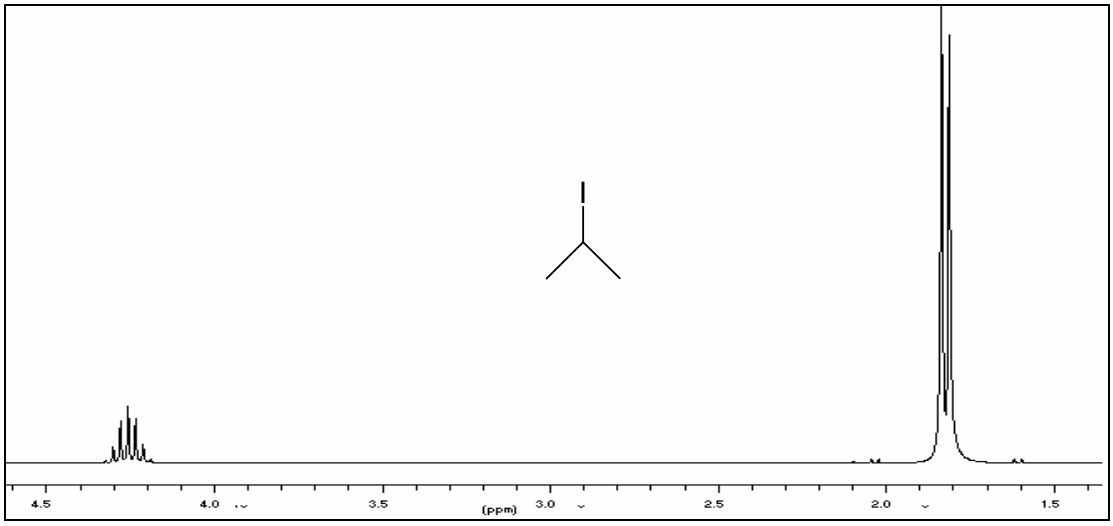
## Analytics

### Reaction monitoring

Stopping the reaction to take a sample lowers the final yield! Monitoring the reaction can be performed by IR spectrocopy.

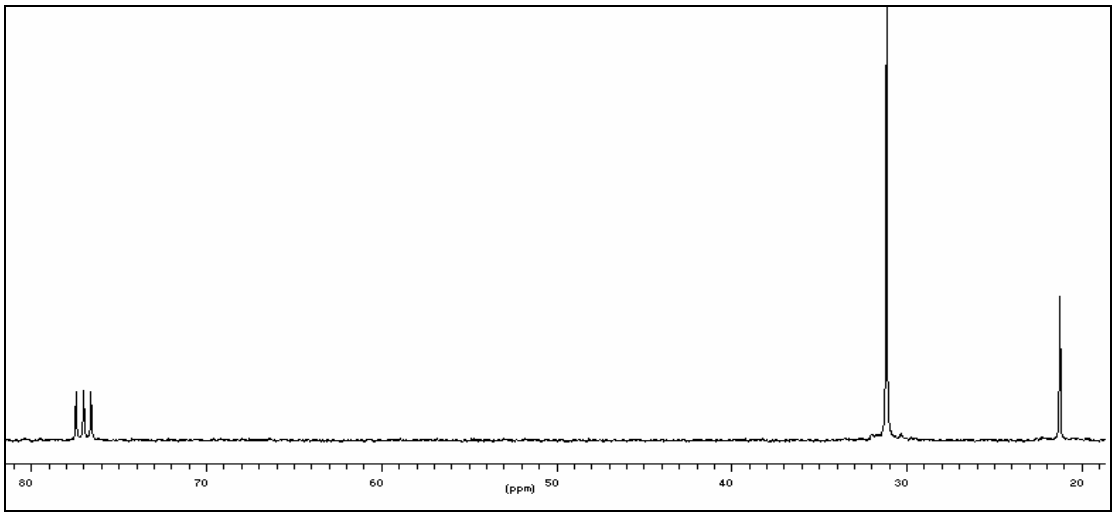
Sample preparation:
1 mL of the reaction mixture is taken with a pipette and distilled with a micro distillation apparatus. The distilled product is used for IR spectroscopy without solvent.
The disappearance of the OH-band at ≈3400 $cm^{-1}$ is an indication of the completeness of the reaction

**$^1$H NMR spectrum of the pure product** (500 MHz, $CDCl_3$)



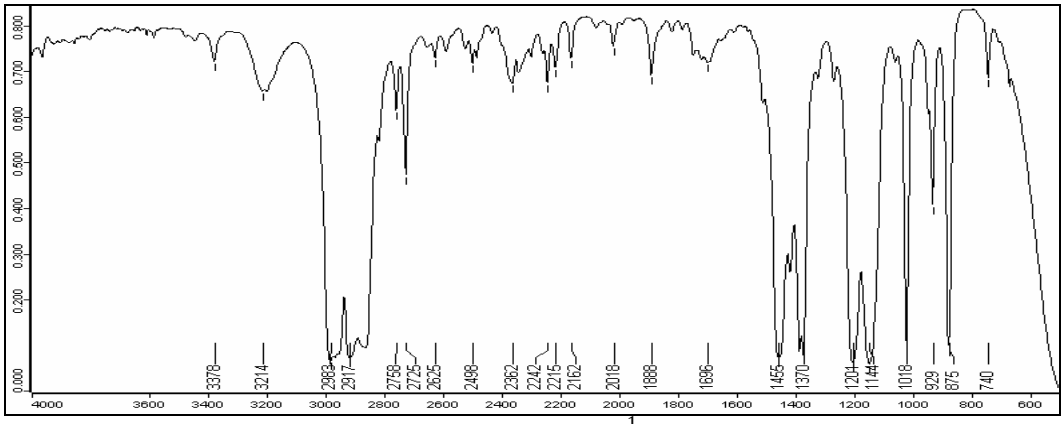

| δ (ppm) | Multiplicity | Number of H | Assignment |
|---|---|---|---|
| 1.82 | d | 6 | $CH_3$ |
| 4.26 | m | 1 | I-CH |

**$^{13}$C NMR spectrum of the pure product** (75.5 MHz, $CDCl_3$)




| δ (ppm) | Assignment |
|---|---|
| 31.13 | **C**$H_3$ |
| 21.23 | I-CH |

**IR spectrum of the pure product** (film)




| (cm$^{-1}$) | Assignment |
|---|---|
| 2983, 2917 | C-H-valence, alkane |
| 1455 | C-H-deformation |
| 1370 | C-H-deformation, -$CH_3$ symm. |