

# Synthesis of tin(IV) iodide

## Summary of the analysed protocols

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| $\text{Sn} + 2\text{I}_2 \rightarrow \text{SnI}_4$<br>$\text{SnI}_2 + \text{I}_2 \rightarrow \text{SnI}_4$<br>$2\text{SnCl}_2 \cdot 2\text{H}_2\text{O} + 2\text{I}_2 \rightarrow \text{SnI}_4 + \text{SnCl}_4 + 2\text{H}_2\text{O}$   | (1)<br>(2)<br>(3) |
| <b>Protocol A<sup>1</sup></b>   |                   |
| <b>Reaction (R<sub>1</sub>):</b> equation (1), stoichiometric proportions of tin and iodine, acetic anhydride and glacial acetic acid (solvents), reflux, T > 100 °C<br><b>Isolation (I<sub>1</sub>):</b> cooling (ice bath) → filtration (suction) → washing (cold chloroform) → drying (air)<br><b>Purification:</b> not prescribed   |                   |
| <b>Protocol B<sup>2</sup></b>   |                   |
| <b>Reaction (R<sub>1</sub>):</b> ≡ Pr A<br><b>Isolation (I<sub>2</sub>):</b> cooling (ice bath) → filtration (suction)<br><b>Purification (Pu<sub>1</sub>):</b> recrystallization – dissolution (chloroform) → drying (desiccator)  |                   |
| <b>Protocol C<sup>3,4</sup></b>   |                   |
| <b>Reaction (R<sub>1</sub>):</b> ≡ Pr A (scale decreased 2.5 times)<br><b>Isolation (I<sub>3</sub>):</b> cooling → filtration (suction)<br><b>Purification (Pu<sub>1</sub>):</b> ≡ Pr B   |                   |
| <b>Protocol D<sup>5</sup></b>   |                   |
| <b>Reaction (R<sub>1</sub>):</b> ≡ Pr C<br><b>Isolation (I<sub>3</sub>):</b> ≡ Pr C<br><b>Purification (Pu<sub>2</sub>):</b> recrystallization – dissolution (chloroform) → drying (air)  |                   |
| <b>Protocol E<sup>6</sup></b>   |                   |
| <b>Reaction (R<sub>2</sub>):</b> equation (1), 17% exc. iodine, anhydrous calcium chloride, acetic anhydride and glacial acetic acid (auxiliary substances), reflux, T > 100 °C<br><b>Isolation (I<sub>3</sub>):</b> ≡ Pr C<br><b>Purification (Pu<sub>3</sub>):</b> recrystallization – dissolution (chloroform) → cooling (ice bath) → filtration → drying (desiccator)         |                   |
| <b>Protocol F<sup>7</sup></b>   |                   |
| <b>Reaction (R<sub>3</sub>):</b> equation (1), 114% exc. tin, acetic anhydride and glacial acetic acid (solvents), reflux, T > 100 °C<br><b>Isolation (I<sub>4</sub>):</b> cooling → filtration (suction) → washing (glacial acetic acid) → drying (suction)<br><b>Purification:</b> not prescribed   |                   |
| <b>Protocol G<sup>8</sup></b>   |                   |
| <b>Reaction (R<sub>4</sub>):</b> equation (1), 114% exc. tin, ethyl acetate (solvent), T < 100 °C<br><b>Isolation (I<sub>5</sub>):</b> cooling (ice bath) → filtration (suction) → washing (cold ethyl acetate) → drying (air)<br><b>Purification:</b> not prescribed   |                   |
| <b>Protocol H<sup>9</sup></b>   |                   |
| <b>Reaction (R<sub>5</sub>):</b> equation (1), 28% exc. tin, chloroform (solvent), reflux, T < 100 °C<br><b>Isolation (I<sub>6</sub>):</b> cooling (ice bath) → heating<br><b>Purification:</b> not prescribed  |                   |
| <b>Protocol I<sup>10-12</sup></b>   |                   |
| <b>Reaction (R<sub>6</sub>):</b> equation (1), stoichiometric proportions of tin and iodine, dichloromethane (solvent), reflux, T < 100 °C<br><b>Isolation (I<sub>7</sub>):</b> filtration (gravity) → washing (hot dichloromethane) → heating → cooling (ice bath) → filtration (suction) → washing (cold dichloromethane) → drying (air)<br><b>Purification:</b> not prescribed |                   |

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| <b>Protocol J<sup>13</sup></b>   |
| <b>Reaction (R<sub>7</sub>):</b> equation (1), 328% exc. tin, dichloromethane (solvent), reflux, T < 100 °C<br><b>Isolation (I<sub>8</sub>):</b> filtration (gravity)<br><b>Purification (Pu<sub>4</sub>):</b> recrystallization – dissolution (dichloromethane) → heating (steam bath) → cooling (ice bath) → filtration (suction) → drying (air)                   |
| <b>Protocol K<sup>14</sup></b>   |
| <b>Reaction (R<sub>8</sub>):</b> equation (1), 328% exc. tin, naphtha VMP (solvent), reflux, T > 100 °C<br><b>Isolation (I<sub>9</sub>):</b> filtration (suction) → washing (naphtha VMP) → cooling → filtration (suction) → drying (suction)<br><b>Purification:</b> not prescribed   |
| <b>Protocol L<sup>15</sup></b>   |
| <b>Reaction (R<sub>9</sub>):</b> equation (1), 29% exc. tin, carbon tetrachloride (solvent), reflux, T < 100 °C<br><b>Isolation (I<sub>10</sub>):</b> filtration → washing (boiling carbon tetrachloride) → cooling (ice bath) → filtration → evaporation of the solvent → cooling<br><b>Purification (Pu<sub>5</sub>):</b> recrystallization (carbon tetrachloride) |
| <b>Protocol M<sup>16</sup></b>   |
| <b>Reaction (R<sub>10</sub>):</b> ≡ Pr L (but different proportion of solvent)<br><b>Isolation (I<sub>10</sub>):</b> ≡ Pr L (scale decreased 3 times)<br><b>Purification (Pu<sub>5</sub>):</b> ≡ Pr L  |
| <b>Protocol N<sup>17</sup></b>   |
| <b>Reaction (R<sub>11</sub>):</b> equation (1), 71% exc. tin, toluene (solvent), T > 100 °C<br><b>Isolation (I<sub>11</sub>):</b> filtration → cooling (ice bath) → filtration (suction) → drying (suction)<br><b>Purification (Pu<sub>6</sub>):</b> recrystallization – dissolution (toluene) → washing (cold toluene)  |
| <b>Protocol O<sup>18</sup></b>   |
| <b>Reaction (R<sub>11</sub>):</b> ≡ Pr N<br><b>Isolation (I<sub>12</sub>):</b> filtration → cooling (ice bath) → filtration (suction) → washing (cold toluene) → drying (desiccator)<br><b>Purification (Pu<sub>7</sub>):</b> recrystallization (cold toluene)   |
| <b>Protocol P<sup>19</sup></b>   |
| <b>Reaction (R<sub>12</sub>):</b> equation (1), stoichiometric proportions of tin and iodine, toluene and anhydrous calcium chloride (auxiliary substances), reflux, T > 100 °C<br><b>Isolation (I<sub>13</sub>):</b> filtration → cooling → washing (toluene) → drying (suction)<br><b>Purification:</b> not prescribed   |
| <b>Protocol Q<sup>18</sup></b>   |
| <b>Reaction (R<sub>13</sub>):</b> equation (2), stoichiometric proportions of tin(II) iodide and iodine, nitrogen and toluene (auxiliary substances), reflux, T > 100 °C<br><b>Isolation (I<sub>14</sub>):</b> filtration → cooling (ice bath)<br><b>Purification (Pu<sub>7</sub>):</b> ≡ Pr O   |
| <b>Protocol R<sup>4</sup></b>  |
| <b>Reaction (R<sub>14</sub>):</b> equation (3), stoichiometric proportions of tin(II) chloride dehydrate and iodine, acetic anhydride and glacial acetic acid (solvents), reflux, T > 100 °C<br><b>Isolation (I<sub>3</sub>):</b> ≡ Pr C<br><b>Purification (Pu<sub>1</sub>):</b> ≡ Pr B   |

<sup>a</sup> → – Sequential

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