A Useful Preparation of *N*-Alkylthio- and *N*-Arylthioimides using Arylthio- and Alkylthiostannanes[1]

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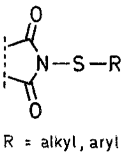
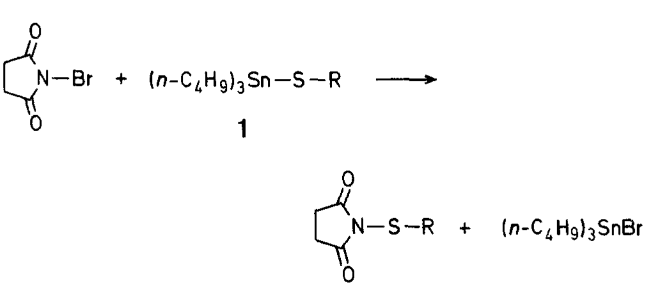
For some years now, *N*-alkylthio- and *N*-arylthioimides of the general type

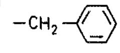
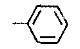


have been found to be very useful as reagents for organothiogroup transfer[2]. While different methods are available for their preparation, not all are applicable for every synthetic situation[3–6]. We here report a reaction for their synthesis which is rapid, general for alkyl and aryl groups, and proceeds in high overall yield making use of alkylthio- and arylthiostannes, compounds which have seen little use for synthetic purposes.

The alkylthio- and arylthiotributylstannanes (**1**) are conveniently prepared in yields of ~ 90 % from the corresponding thiols and tributyltin chloride[7]. The coupling reaction of **1** with either *N*-chloro- or *N*-bromosuccinimide to give **2** takes place in overall yields averaging 90 %.


1

2

1,2	R
a	
b	
c	C_2H_5
d	n-$C_{10}H_{21}$

The advantages of the method are the ready availability of both starting materials and the fact that no disulfide is formed as a by-product. The formation of disulfide is a drawback in a number of different published schemes for the preparation of the title compounds[3,4]; as a consequence, yields are often lower and product isolation more difficult. There are other methods for the preparation of *N*-alkylthio- and *N*-arylthioimides (**2**); however, yields are often low or not reported[5,6]. In sum, this method provides *consistent* yields of isolated product of ~ 90 %. Literature methods give comparable yields for some structural types but lower for others.

Table. Alkylthio- and Arylthiosuccinimides (**2**) prepared

2	Yield[a] [%]	m.p. [°C] or b.p. [°C]/torr	Molecular Formula or Lit. Data [°C]
a	94	m.p. 165–166° (chloroform/hexane)	m.p. 165–166°[5]
b	80	m.p. 114–115° (chloroform/hexane)	m.p. 116°[5]
c	90	m.p. 44–45° (ether/hexane)	m.p. 48–49°[4]
d	89[b]	b.p. 180°/0.5 m.p. 45–46° (solvent)	$C_{14}H_{25}NO_2S$ (271.4)

[a] Yields reported are of isolated material for several runs.

[b] The purity of this product was satisfactory according to T.L.C. The M.S. and ^{1}H-N.M.R. spectra were consistent with the structure.

	C	H	N
calc.	61.96	9.28	5.16
found	62.21	9.05	4.83

***N*-Alkylthio- and *N*-Arylthiosuccinimides (2); General Procedure:** To a stirred suspension of *N*-bromosuccinimide (1.42 g, 8 mmol) in dichloromethane (10 ml) at 0 °C is added a solution of the alkylthio- or arylthiotributylstannane (**1**; 10 mmol) in dichloromethane (10 ml). The solution immediately changes color from light yellow to red, depending on the nature of the group R. The reaction mixture is stirred for 1 h during which time the color disappears. The solution is then diluted with dichloromethane (20 ml), washed with aqueous 5% potassium hydroxide (20 ml), and dried. The solvent is evaporated and the remaining crude product **2** either distilled in vacuo, or stirred with hexanes and the resultant crystalline materials recrystallized.

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