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Alexander Senning[a]
[a] Kemisk Institut, Aarhus Universitet, Århus C, Denmark

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INTERESTING ERRORS IN SULFUR CHEMISTRY, 11

ALEXANDER SENNING
Kemisk Institut, Aarhus Universitet, DK-8000 Århus C, Denmark

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DIVINYL DISULFIDE

For starters, a Chemical Abstracts search for the title compound **1**, $CH_2=CH-S-S-CH=CH_2$, RN [15805-34-2], is beset with many difficulties. Online, a search in the CA file retrieved Ref.[4] plus two false drops, Ref.[5] (which deals with diallyl disulfide) and Ref.[6] (which refers to "ethylene disulfide", $(C_2H_4SS)_n$). A subsequent search in the CAOLD file provided Ref.[2] Thus, Refs.[1,3] can only be found in the paper edition of Chemical Abstracts.

The oldest reported synthesis of **1** is from 1957 and based on the dehydrochlorination of bis(2-chloroethyl) disulfide with ethanolic potassium hydroxide or other strong bases. Compound **1** is obtained as an orange solid, m.p. 20 °C, b.p. 180–183 °C (with incipient decomposition). A satisfactory elemental analysis is performed. A homopolymer of **1** as well as a copolymer of **1** with styrene are also described.[2]

In a later synthetic procedure (from 1969) **1** is made by oxidation of ethenethiolate anion with iodine and described as a liquid, b.p. 38–42 °C/10 mm Hg, n_D^{20} 1.561, which "polymerizes exothermally at room temperature". "Even from a 10% solution in ether solid material precipitates within 1 hr". The authors record a satisfactory ^1H NMR spectrum, but find **1** too unstable for mass spectrometry and elemental analysis. No mention of Ref.[2] is made.[3]

In a subacute inhalation toxicity study carried out in 1979 **1**, b.p. 86 °C, is used as "submitted by the manufacturing divisions of ICI Ltd." and a provisional operational limit of 2 ppm is established.[4]

The very oldest mention of **1** stems from 1951 and describes its reaction with ammonium hypophosphite in the presence of di-*t*-butyl peroxide at 120 °C for 8 h to form the corresponding mono-and diphosphinates. Neither the source nor the physical properties of **1** are disclosed.

It seems that the majority of reports mentioning **1** must be in error and that the error sources remain elusive.

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