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SULFOLANE AS A NATURAL PRODUCT FROM THE SPONGE/TUNICATE COMPOSITE, *BATZELLA* SP./*LISSOCLINUM* SP.

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ABSTRACT.—An inseparable composite consisting of the sponge *Batzella* sp. and the tunicate *Lissoclinum* sp. collected from Point Impossible on the southern coast of Victoria, Australia, was found to contain sulfolane (**1**), as confirmed by spectroscopic analysis and comparison with an authentic sample.

In our continuing investigations into the chemistry of southern Australian marine organisms our attention was drawn to a specimen that was subsequently identified as a composite between a sponge (*Batzella* sp.) and a tunicate (*Lissoclinum* sp.). The creamy/pink encrusting sponge/tunicate (1–3 mm thick and 1–8 cm diameter) was collected on two separate occasions, February 1990 (17 g dry wt) and November 1990 (34 g dry wt), from a shallow reef exposed only at extreme low tides, at Point Impossible, Victoria, Australia. After collection the specimens were placed in EtOH and stored at -20° until required.

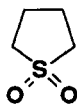
The decanted EtOH extract was concentrated under reduced pressure to yield a yellow gum that was triturated with CH_2Cl_2 , and the resulting concentrate was subjected to rapid silica filtration (10% stepwise elution from hexane to EtOAc). The most polar fraction was further fractionated by hplc (Phenomenex spherex 5μ silica 250×10 mm eluted with 75% EtOAc/hexane) to yield pure sulfolane (**1**) (Synonyms include tetrahydrothiophene-1,1-dioxide and tetramethylene sulfone) (Feb. 0.8 mg, 0.0047%; Nov. 1.8 mg, 0.0053%). Spectroscopic data accumulated on the isolated material were identical with

those of authentic sulfolane. Similarly, co-injection of isolated and authentic samples of sulfolane onto both capillary gc and normal phase hplc resulted in identical retention times. As **1** was found in two quite separate collections, made some 10 months apart and processed with entirely different batches of solvent, we can discount the possibility that sulfolane is a contaminant introduced during the isolation process. Consistent with this is the observation that sulfolane has never been isolated from any other specimens in our laboratory, either before, during, or after this investigation. Although the known chemistry of *Batzella* sponges and *Lissoclinum* tunicates is dominated by sulfur-containing metabolites (1–3), to the best of our knowledge this report represents the first occasion, either terrestrial or marine, where sulfolane (**1**) has been described as a natural product.

EXPERIMENTAL

GENERAL EXPERIMENTAL PROCEDURES.—General procedures were as described by Butler *et al.* (4).

SPONGE/TUNICATE COMPOSITE.—Most of the specimen is an encrusting compound tunicate, *Lissoclinum* sp. (Didemnidae), with bits of sponge, *Batzella* sp. (Poesilosclerida: Desmacididae), dispersed around the test. The sponge consists of a thin layer of spongin fiber lying under the tunicate and hence on the substrate. The specimen incorporates a dense layer of styles embedded within the basal spongin, standing perpendicular to the substrate. The styles measure 460–625 μm long and 8–24 μm wide. No other spicules or collagenous spongin were observed. A specimen has been lodged with the

**1**

collection at the Northern Territory Museum and has been assigned the registry number Z3721. Numerous individual specimens were collected during both the February 1990 and November 1990 collection trips. Individual specimens were found as creamy/pink encrustations ca. 1–3 mm thick and 1–8 cm in diameter on the under surface of rocks and reef overhangs. Collection was only feasible at time of extreme low tide when the reef flats were exposed. At most other times the reef flats were prone to significant wave and surf action that made collection extremely difficult and hazardous.

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