

was washed with H₂O, dried (MgSO₄), and concd. The yield of crude product was 89% (C=O, 1780 cm⁻¹). The chloroformate was converted into the desired carbamate by adding 1 equiv each of an amine (ethylenimine or Me₂NH) and Et₃N. The conditions and work-up procedure paralleled those just described.

Citronellol was converted into the P derivatives by treatment with 1 equiv each of the required acid halide and Et₃N.

Citronellylamine Derivatives.—The amine was prepared by LAH reduction of the oxime.⁸ However, yields were variable and depended largely on the freshness of the reducing agent. Citronellylamine reacted with hexamethylene diisocyanate in Et₂O exothermically to produce the diurea as a ppt. The amine was converted into its other listed derivatives by reaction with the acid halide and Et₃N as described for citronellol.

Epoxidation of the ethyl (3,7-dimethyl-6-octenyl)carbamate was carried out with 1 equiv of *m*-chloroperbenzoic acid in CH₂Cl₂. The mixture was held at 5–10° during addn of the olefins and was allowed to stand at room temp overnight. After extraction with Na₂CO₃, the organic soln was dried (Na₂SO₄), concd, and dist by using a short-path distn apparatus.

The nmr spectrum of *P,P*-bis(1-aziridinyl)-*N*-(3,7-dimethyl-6-octenyl)phosphinic amide showed the following absorptions: 0.88 d (CH₃CH), 1.57 and 1.65 (cis and trans allylic Me, respectively), 1.93 d ($-\overset{\text{N}}{\underset{\text{P}}{\text{C}}}-$, $J_{\text{PN}} = 15 \text{ Hz}$), 5.05 (vinyl H).

Acknowledgment.—The authors express their gratitude to C. G. LaBrecque and coworkers, Entomology Research Division's Insects Affecting Man Investigations Laboratory, Gainesville, Fla., for the tests of housefly sterility.

(8) D. Arigoni and O. Jeger, *Helv. Chim. Acta.*, **37**, 881 (1954).

Synthesis of New Urethans. *p*-Ethylsulfonyl- and *p*-Dimethylsulfamoylcarbanilic Acid Esters

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Received April 23, 1970

Several *p*-arylsulfonylcarbanilic acid esters were reported to have antitumor activities.¹ New urethans listed in Table I were prepared by Curtius degradation of appropriate benzoyl azides.

The compounds proved to be inactive² (*T/C* = 89–102% at 400 mg/kg) against the L-1210 lymphoid leukemia in BDF₁ mice, and the Walker carcinoma 256 in random-bred albino rats.

Experimental Section³

***p*-Ethylsulfonylbenzoyl Azide.**—*p*-Ethylsulfonylbenzoic acid ethyl ester (mp 64°) was prepared by known methods from *p*-ethylsulfonylbenzoic acid⁴ and transformed to *p*-ethylsulfonylbenzoylhydrazide (mp 164°). This hydrazide (2.28 g, 0.01 mole) in 20 ml of 50% AcOH was stirred vigorously at ice bath temperature to give 2.27 g of azide (95%) mp 125° dec. *Anal.* (C₉H₉N₃O₃S) C, H, N.


p-Dimethylsulfamoylbenzoyl azide was prepared similarly from *p*-dimethylsulfamoylbenzoyl hydrazide⁵ as a white powder (91%), mp 109° dec. *Anal.* (C₉H₁₀N₄O₃S) C, H, N.

(1) Al. Mavrodin, C. Demetrescu, and C. Chirita, *Rev. Roum. Chim.*, **10**, 1025 (1965).

(2) Screening results were supplied by CCNCS of the National Institutes of Health, Bethesda, Md.

(3) Melting points were taken on a Kofler hot stage microscope and were uncorrected. The ir spectra were determined with a Leitz model 111 spectrograph. Nmr spectra were obtained on a Varian A60A instrument.

(4) H. Sato, *Yakugaku Zasshi*, **72**, 74 (1952).

TABLE I
RSO₂  NHCOOR'

R	R'	Mp, °C	Yield, %	Formula ^a
Et	Et	135	89	C ₁₁ H ₁₅ N ₂ O ₄ S
Et	<i>t</i> -Bu	129	93	C ₁₃ H ₁₉ N ₂ O ₄ S
Et	<i>n</i> -Hexyl	109	95	C ₁₅ H ₂₃ N ₂ O ₄ S
Et	<i>n</i> -Octyl	124	88	C ₁₇ H ₂₇ N ₂ O ₄ S
Et	Allyl	121	86	C ₁₂ H ₁₅ N ₂ O ₄ S
Et	Benzyl	136	96	C ₁₆ H ₁₇ N ₂ O ₄ S
Et	Cholesteryl	222	92	C ₃₆ H ₅₅ N ₂ O ₄ S
Et	Cyclopentyl	142	79	C ₁₄ H ₁₉ N ₂ O ₄ S
Et	Cyclohexyl	139	83	C ₁₅ H ₂₁ N ₂ O ₄ S
Et	Cycloheptyl	120	85	C ₁₆ H ₂₃ N ₂ O ₄ S
Et	Cyclooctyl	127	79	C ₁₇ H ₂₅ N ₂ O ₄ S
Et	<i>o</i> -MeOC ₆ H ₄	191	80	C ₁₅ H ₁₇ N ₂ O ₆ S
Et	Thymyl	143	84	C ₁₉ H ₂₃ N ₂ O ₄ S
Et	6-Allyl-4-MeOC ₆ H ₃	154	73	C ₁₉ H ₂₁ N ₂ O ₆ S
Et	Ph ₂ CH	195	76	C ₂₂ H ₂₁ N ₂ O ₄ S
Et	α -Cyclohexyl- α -methylbenzyl	136	79	C ₂₃ H ₂₉ N ₂ O ₄ S
Et	<i>p</i> -Menth-3-yl	170	92	C ₁₉ H ₂₉ N ₂ O ₄ S
Me ₂ N	Et	127	71	C ₁₁ H ₁₆ N ₂ O ₄ S
Me ₂ N	<i>i</i> -Pr	159	76	C ₁₂ H ₁₈ N ₂ O ₄ S
Me ₂ N	<i>t</i> -Bu	169	78	C ₁₃ H ₂₀ N ₂ O ₄ S
Me ₂ N	<i>n</i> -Am	105	72	C ₁₄ H ₂₂ N ₂ O ₄ S
Me ₂ N	<i>n</i> -Hexyl	95	69	C ₁₅ H ₂₄ N ₂ O ₄ S
Me ₂ N	<i>n</i> -Octyl	107	68	C ₁₇ H ₂₈ N ₂ O ₄ S
Me ₂ N	Allyl	110	73	C ₁₂ H ₁₆ N ₂ O ₄ S
Me ₂ N	Cyclopentyl	170	86	C ₁₄ H ₂₀ N ₂ O ₄ S
Me ₂ N	Cyclohexyl	176	81	C ₁₅ H ₂₂ N ₂ O ₄ S
Me ₂ N	Cycloheptyl	170	83	C ₁₆ H ₂₄ N ₂ O ₄ S
Me ₂ N	Cyclooctyl	174	79	C ₁₇ H ₂₇ N ₂ O ₄ S
Me ₂ N	Benzyl	142	88	C ₁₆ H ₁₈ N ₂ O ₄ S
Me ₂ N	Cholesteryl	220	80	C ₃₆ H ₅₆ N ₂ O ₄ S
Me ₂ N	Ph ₂ CH	195	91	C ₂₂ H ₂₂ N ₂ O ₄ S
Me ₂ N	Ph ₃ C	132	65	C ₂₃ H ₂₆ N ₂ O ₄ S
Me ₂ N	Thymyl	155	78	C ₁₉ H ₂₄ N ₂ O ₄ S
Me ₂ N	<i>p</i> -Menth-3-yl	173	76	C ₁₉ H ₃₀ N ₂ O ₄ S
Me ₂ N	<i>o</i> -Methoxyphenyl	155	69	C ₁₆ H ₁₈ N ₂ O ₅ S
Me ₂ N	6-Allyl-4-MeOC ₆ H ₃	193	70	C ₁₉ H ₂₂ N ₂ O ₅ S
Me ₂ N	α -Cyclohexyl- α -methylbenzyl	120	64	C ₂₃ H ₃₀ N ₂ O ₄ S

^a All compounds were analyzed for C, H, N, and the results were satisfactory. Similarly ir and nmr spectra were as expected.

***p*-Ethylsulfonylcarbanilic Acid Benzyl Ester.**—*p*-Ethylsulfonylbenzoyl azide, (2.3 g, 0.01 mole) and 2.48 g (0.02 mole) of benzyl alcohol was refluxed for 1 hr in 20 ml of dry PhMe. The solvent was evaporated and the residue was recrystallized from dil EtOH to give 2.9 g (90%) of white plates, mp 136°. The other compounds listed in Table I were prepared in a similar way, except for the Et esters which were prepared by 3-hr refluxing of the appropriate azide in 10 times its weight of abs EtOH.

(5) H. Shirai, M. Yoneda, and N. Oda, *Nagoya Shiatsu Daigaku, Yakugaku Kyo.* **2**, 45 (1954); *Chem. Abstr.*, **50**, 11337 (1956).

Synthesis of 5,7-Dimethoxyindole¹¹

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Received April 6, 1970

The predominance of the indole nucleus and its methoxy analogs in many biologically active systems

¹¹ This work was supported by NASA Grant NGR 05-029-006.