(activity II–III) basic alumina. The benzene eluate afforded 0.190 g (56%) of a light yellow oil.

The product underwent slight decomposition upon distillation [short path, oven temperature 140° (0.1 mm)] or upon preparative thick layer chromatography with silica gel H: λ_{max} (film) 3.0, 3.45, 3.55, 5.90, 6.10, 6.92, 7.30, 9.05, 10.60, 11.0, and 13.25 mμ; δ_{TMS} ($CDCl_3$) 1.08 ppm (s, CH_3). After standing for several weeks a sample solidified (mp 77–78°) but satisfactory analytical values could not be obtained.

***t*-2-Methyl-*r*-1H,*c*-5H,*c*-7H,*t*-8H-tricyclo[6.3.0.0^{5,7}]undecan-*c*-2-ol (11).** A mixture of 100 mg (0.55 mmol) of the dibromohydroazulenol **10**, 100 mg of lithium wire, and 3 ml of *tert*-butyl alcohol in 3 ml of tetrahydrofuran was stirred at room temperature for 2 days.[16] The mixture was cautiously quenched with 12 ml of water and the product was isolated with ether. Short-path distillation [oven temperature 70°(0.1 mm)] afforded 50 mg (50%) of a colorless oil, which gave a predominant peak on the gas chromatogram (UCW-98 column at 205°) with retention time of 3.2 min: λ_{max} (film) 3.0, 3.45, 6.9, 7.3, 9.0, 11.0, and 13.75 mμ; δ_{TMS} ($CDCl_3$) 0.68 (m, cyclopropyl H) and 1.10 ppm (s, CH_3).

Anal. Calcd for $C_{12}H_{20}O$: C, 79.94; H, 11.18. Found: C, 79.68; H, 11.19.

***t*-2-Methyl-*r*-1H,*t*-5H,*t*-7H,*t*-8H-tricyclo[6.3.0.0^{5,7}]undecan-*c*-7-ol (9).** A mixture of 0.332 g (2 mmol) of the hydroazulenol **8**,[1] 3.42 g (6.5 mmol) of methylene iodide, and 1.4 g (20 mmol) of zinc–copper couple in 20 ml of ether was stirred at room temperature for 3.5 days.[8] The mixture was filtered, and the ether was washed with 3% aqueous hydrochloric acid. Work-up of the ether solution and short-path distillation [oven temperature 70° (0.1 mm)] of the product afforded 0.350 g (97%) of a colorless oil which showed a predominant peak on the gas chromatogram (UCW-98 column at 205°) with retention time of 3.8 min: λ_{max} (film) 3.0, 3.45, 6.95, 7.35, 9.20, 9.82, 10.62, 11.05, and 13.75 mμ; δ_{TMS} ($CDCl_3$) 0.44 (m, cyclopropyl H) and 1.10 ppm (s, CH_3).

Anal. Calcd for $C_{12}H_{20}O$: C, 79.94; H, 11.18. Found: C, 79.95; H, 11.31.

A sample of this material solidified (mp 43–44°) upon short-path distillation.

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Registry No.—**1**, 51310-36-2; **2**, 51310-37-3; **4**, 51371-46-1; **5**, 51310-38-4; **6**, 51310-39-5; **7**, 51371-47-2; **8**, 30166-33-7; **9**, 51310-40-8; **10**, 51310-41-9; **11**, 51371-48-3.

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(11) We are indebted to Professor G. Büchi for a sample of natural globulol.
(12) The apparatus described by W. S. Johnson and W. P. Schneider ("Organic Syntheses," Collect. Vol. IV, Wiley, New York, N. Y., 1963, p 132) was used to maintain an argon atmosphere. The isolation procedure consisted of thorough extractions with the specified solvent, washing the combined extracts with saturated brine solution, and drying the extracts over anhydrous magnesium sulfate. The solvent was removed from the filtered extracts under reduced pressure on a rotary evaporator. Microanalyses were performed by Micro-Tech Laboratories, Inc., Skokie, Ill. Infrared spectra were obtained with a Perkin-Elmer 137 spectrophotometer. Infrared absorptions are reported in wavelengths (mμ) and are standardized with reference to the 6.24-mμ peak of polystyrene. Nuclear magnetic resonance spectra were recorded with a Varian T-60 spectrometer. Signals are reported as the chemical shift downfield from tetramethylsilane (TMS) in parts per million (ppm) of the applied field. The multiplicity of the peak is abbreviated: singlet, s; doublet, d; triplet, t; quartet, q; and multiplet, m. Coupling constants are reported in hertz (Hz). Melting points were determined on a calibrated Thomas capillary melting point apparatus. Melting points are not corrected.
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Preparation of Azetidine and Some *N*-Aroylazetidines[1]

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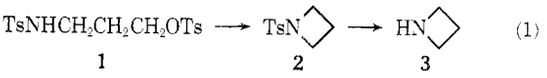
In the course of examination of selected amides of potential value in the Vilsmeier–Haack reaction it became evident that little information is available on otherwise unsubstituted *N*-aroylazetidines. Only a few of these compounds appear to have been reported.[2,3] Moreover, existing methods for the preparation[4-7] of azetidine itself are not only lacking in experimental simplicity but are unsuitable when it is desired to prepare small quantities only.

Investigation of certain of the reactions reported indicated that the sequence employed by Vaughan, *et al.* (eq 1) could, by the application of improved methods, be used



1 2 3 (1)

in small-scale operations to afford a somewhat better yield than previously reported.[5] Thus, the cumbersome cyclization of **1** by the action of ethoxide or *tert*-butoxide over 16 hr was found to be unnecessary, as the same result could be obtained by use of aqueous hydroxide over approximately 4 hr with much less effort.

For the reductive detosylation of **2**, sodium naphthalenide[8] in diglyme medium (which in the present context is far superior to dimethoxyethane or tetrahydrofuran) was found to be suitable. The benefit of this method lies not only in considerably improved yields but also in the greater simplicity of operation than that of Vaughan's method where reduction by sodium in amyl alcohol affords some 40% of product only after a somewhat tedious sequence of operations.

The present radical-anion reduction is quick and avoids complex work-up procedures, as the azetidine is obtained as an approximately 1 *M* solution in diglyme by simple distillation from the reaction mixture. The yield of azetidine is approximately 70% of theoretical.

The *N*-aroylazetidines listed in Table I were prepared in fair yield by the use of the azetidine–diglyme solution (to which was added an equimolecular quantity of triethylamine in order to conserve azetidine and simplify purification) and the appropriate aroyl chloride. The identity of each of these derivatives was established by the usual instrumental and analytical means.

Table I

ArCON⟨a,b⟩

Ar	Mp,[a] °C	Yield, %	$\nu_{C=O}$, cm^{-1} [b]	δ_{-CH_2-}	Registry no.[c]
Phenyl	61–62	54	1625	a 4.31 b 2.35	3420-62-0
4-Nitrophenyl	122–123	56	1620	a 4.36 b 2.45	51425-89-9
4-Chlorophenyl	107.0	45	1605	a 4.30 b 2.36	51425-90-2
4-Tolyl	86–86.5	68	1605	a 4.30 b 2.34	51425-91-3
3,5-Dinitrophenyl	155–156	48	1630	a 4.47 b 2.53	51425-92-4
2-Furyl	120–120.5	65	1620	a 4.42 b 2.40	51425-93-5

[a] Uncorrected, recrystallized from hexane or toluene–hexane. [b] Run as Nujol mulls. [c] Satisfactory analytical data (±0.3% for C, H, Cl, N) were reported for all new compounds listed.

Experimental Section

3-(4-Toluenesulfonamido)propyl-4-toluenesulfonate (1) was prepared by the method of Vaughan, *et al.*,[5] and used without further purification.

4-Toluenesulfonazetidide (2). To a suspension of 6.72 g (17.5 mmol) of 1 in 550 ml of water (the optimum volume for the high-dilution technique required) was added 176 ml of 0.1 *M* sodium hydroxide solution and the mixture was refluxed until most of the solid had dissolved (3–4 hr). The light pink solution was treated with charcoal and filtered while hot. On cooling, glittering white needles, 3.15 g (85% average), mp 123–124° (lit. mp 118–120°), were formed.

Azetidine (3). A solution of 16 g (125 mmol) of naphthalene in 100 ml of diglyme was magnetically stirred in a 500-ml flask fitted with a dropping funnel and a short fractionation column coupled to a condenser, receiver, and three small ice-cooled traps containing diglyme. The apparatus was flushed with dry argon, and *ca.* 10 g of sodium shot was added in one batch. After stirring for 30 min, a solution of 5.28 g (25 mmol) of **2** in 60 ml of warm diglyme was added over 40 min and stirring was continued for a further 30 min under a very slow stream of argon. This was followed by distillation (terminated when the boiling point of diglyme was reached) and the combined distillate (*ca.* 20 ml) and trap contents (*ca.* 10 ml) were assayed acidimetrically. Average yield was 70% of theoretical.

Registry No.—**1,** 51425-88-8; **2,** 7730-45-2; **3,** 503-29-7; benzoyl chloride, 98-88-4; 4-nitrobenzoyl chloride, 122-04-3; 4-chlorobenzoyl chloride, 122-01-0; 4-methylbenzoyl chloride, 874-60-2; 3,5-dinitrobenzoyl chloride, 99-33-2; 2-furancarbonyl chloride, 527-69-5.

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