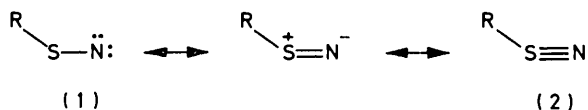


Intermolecular Trapping of Sulphenylnitrenes by Alkenes

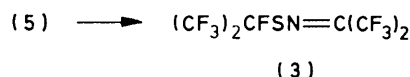
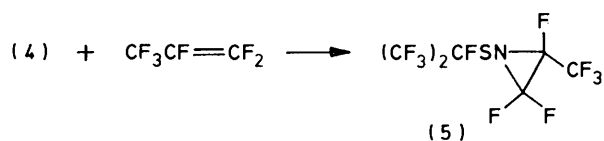
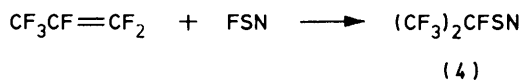
By Robert S. Atkinson* and Brian D. Judkins, Department of Chemistry, Leicester University, Leicester LE1 7RH

Oxidation of 2,4-dinitrobenzenesulphenamide with lead tetra-acetate in the presence of electron-rich alkenes (styrene, (*E*)- and (*Z*)-1-phenylpropene, 2-phenylpropene, (*Z*)-but-2-ene, and butadiene) gives the corresponding substituted *N*-(2,4-dinitrophenylsulphenyl)aziridines. Intermolecular trapping of a presumed sulphenylnitrene intermediate is also successful in the oxidation of 2-nitrobenzenesulphenamide but fails for RSNH_2 when $\text{R} = \text{PhCO}$, 4- ClC_6H_4 , or 4- $\text{NO}_2\text{C}_6\text{H}_4$.

A SULPHENYLNITRENE (1) is one possible resonance contributor to the thiazynone (2). Fluoro-¹ and chloro-thiazynone² (2; $\text{R} = \text{F}, \text{Cl}$) are known compounds which show few reactions characteristic of nitrenes. However,



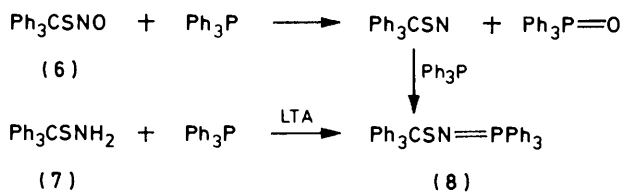
fluorothiazynone reacts with perfluoropropene in the presence of caesium fluoride to give, among other products, the sulphenylimine (3).³ The proposed route to (3) (Scheme 1) involved formation of the perfluoro-



SCHEME 1

sulphenylnitrene (4) and addition of this nitrene to to perfluoropropene followed by rearrangement of the resulting aziridine (5).

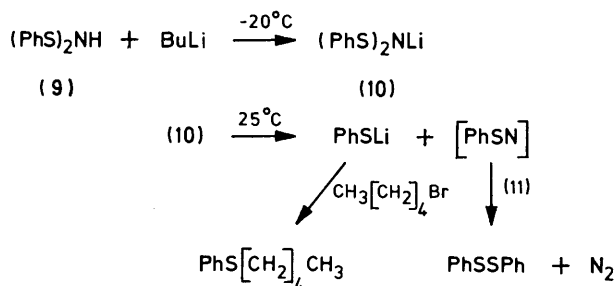
A sulphenylnitrene intermediate has been postulated in the deoxygenation of tritylthionitroso (6) with triphenylphosphine and in the oxidation of triphenylmethanesulphenamide (7) with lead tetra-acetate to give the phosphinimine (8) (Scheme 2).⁴



SCHEME 2

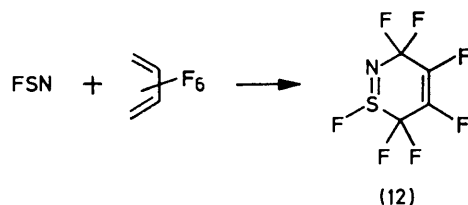
Treatment of the sulphenimide (9) with butyl-lithium at low temperature gave the anion (10) which was reported to be stable up to -28°C but to decay to diphenyl

disulphide above this temperature *via* phenylsulphenylnitrene⁵ (11) (Scheme 3).



SCHEME 3

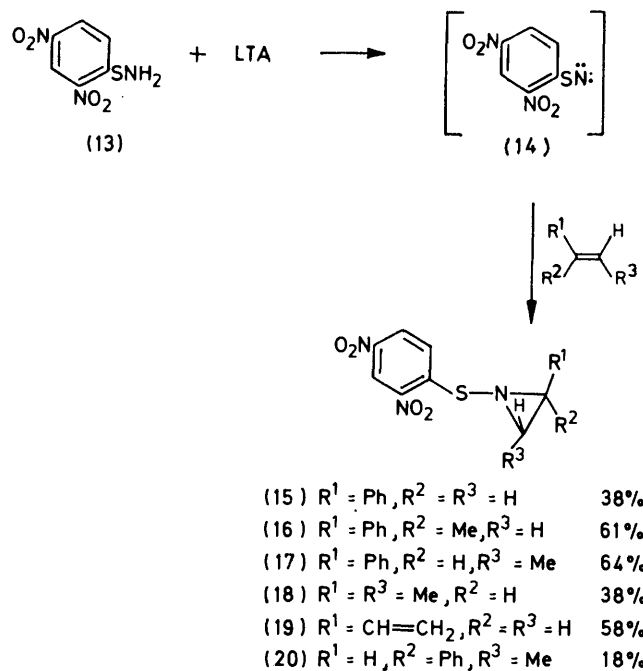
Perhaps the best evidence of sulphenylnitrene character and support for the mechanism proposed in Scheme 1 would be the isolation of aziridines by intermolecular trapping with alkenes. From the limited evidence available, thiazynones (*e.g.* fluorothiazynone) undergo cycloaddition with the $\text{S}\equiv\text{N}$ bond acting as a 2π component. Thus addition of fluorothiazynone to perfluorobutadiene gave the 1,2-thiazacyclohexa-1,4-diene (12).⁶ For aziridine formation, this reactivity must be suppressed in favour of the nitrogen acting as a 2π component in a cheletropic addition to an alkene.



In this paper we report that oxidation of the 2,4-dinitrobenzenesulphenamide (13) with lead tetra-acetate generates the corresponding sulphenylnitrene (14) which has been trapped by alkenes to give aziridines (15)–(20).⁷ It is clear that the nitrene (14) reacts better with the more electron-rich olefins: no aziridines were obtained using methyl acrylate, ethyl cinnamate, 3,3-dimethylbut-1-ene, or 2-acetylbenzofuran as the alkene traps: all of these substrates react with *N*-nitrenes.⁸ The formation of the aziridine (19) from the sulphenylnitrene (14) and butadiene is in contrast to the formation of (12) from fluorothiazynone.

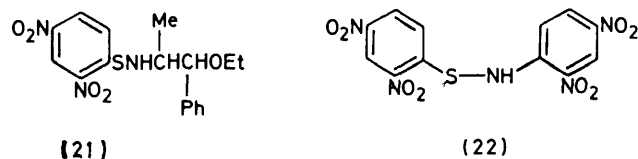
The structures of these aziridines (15)–(20) follow

from their characteristic ring-proton coupling constants and chemical shifts in their n.m.r. spectra. Moreover, the sulphenylaziridines (15) and (17) were reduced by sodium borohydride in ethanol-dichloromethane to the



known 2-phenylaziridine and *trans*-2-methyl-3-phenylaziridine in 44 and 56% yield respectively.

Non-stereospecific addition of the nitrene (14) to *cis*-1-phenylpropene occurred to give a mixture of *cis*- and



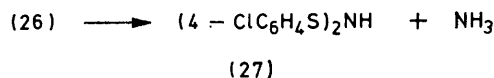
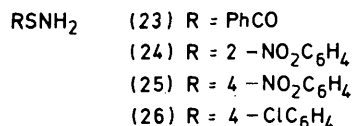
trans-2-methyl-3-phenyl-substituted aziridines (17) and (20) which accounts for the lower yield of (20). Chromatography of the reaction mixture of (17) and (20) [in which (17) predominates] over basic alumina using light petroleum-ethyl acetate allows isolation of the pure *cis*-isomer (20) since the *trans*-isomer (17) is attacked on the

column to give the ring-opened sulphenamide (21). This ring-opening can be reproduced by treatment of (17) with sodium ethoxide in ethanol.

Oxidation of 2,4-dinitrobenzenesulphenamide (13) with lead tetra-acetate in the absence of any trap gave a yellow crystalline material in 40% yield. It had two types of dinitrophenyl ring protons in its n.m.r. spectrum and a mass peak at 381 in its mass spectrum. The assigned structure (22) was confirmed by an unambiguous synthesis of this compound from 2,4-dinitroaniline and 2,4-dinitrobenzenesulphenyl chloride. In dilute base or on a basic alumina column, the acidic N-H in (22) is removed with the production of a blue colour.

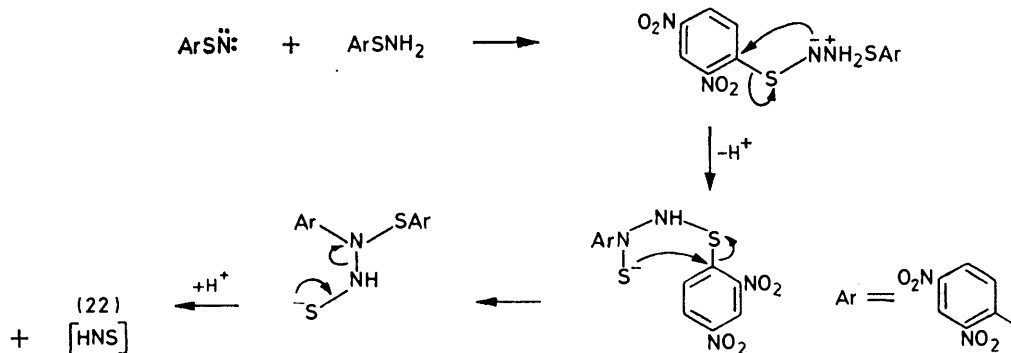
There are a number of mechanisms which can account for the formation of (22) depending on whether attack of the nitrene (14) takes place on the nitrogen or the sulphur of (13). If attack takes place on nitrogen then two consecutive Smiles rearrangements lead to the product (Scheme 4).

Oxidation of Other Sulphenamides RSNH_2 .—The sulphenamides (23), (24), and (25) are readily available compounds. 4-Chlorobenzenesulphenamide (26) was prepared as an unstable oil by amination of 4-chlorobenzenethiol in aqueous base by hydroxylamine *O*-sulphonic acid: it readily disproportionates to the sulphenimide (27) and ammonia on standing at room temperature.



Oxidation of (23)–(26) with lead tetra-acetate in dichloromethane gave evolution of a gas in all cases. In the case of (23), the products were elemental sulphur and an intractable tar. The products from similar oxidation of (24)–(26) were the corresponding disulphides ArSSAr .

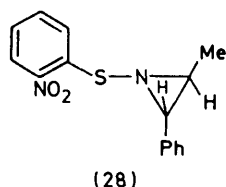
Attempts to trap the nitrenes which are presumably intermediates in oxidations of (23), (25), and (26) were unsuccessful. Thus no identifiable addition products



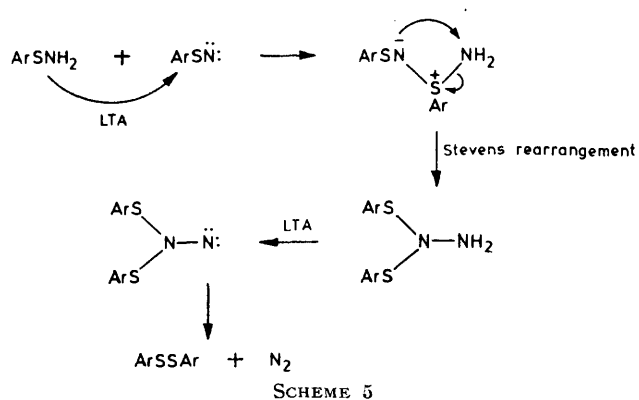
SCHEME 4

were obtained from oxidation of the benzoylsulphenamide (23) in the presence of methyl acrylate, styrene, or 4-chlorophenyl crotyl sulphide.⁹ Oxidation of 4-chlorophenylsulphenamide (26) in the presence of methyl acrylate, *trans*-1-phenylpropene, or buta-1,3-diene gave only bis(4-chlorophenyl) disulphide. Using 4-nitrobenzene sulphenamide (25), no identifiable trapped products were obtained with 4-chlorophenyl crotyl sulphide, styrene, or dimethyl sulphoxide although with *trans*-1-phenylpropene, a very low yield of the expected aziridine was detectable by n.m.r. spectroscopy.

Oxidation of the *o*-nitrobenzenesulphenamide (24) in the presence of *trans*-1-phenylpropene, however, gave aziridine (28) in 58% yield.



Evidently the efficient trapping of these aryl sulphenylnitrenes is very sensitive to the nature of the aryl substituent. This sensitivity may be related to the ability of the unchanged sulphenamide ArSNH_2 to trap the corresponding sulphenylnitrene ArSN^\cdot . We have shown elsewhere that the divalent sulphur in aryl allyl sulphides is an excellent trap for *N*-nitrenes⁹ and 2,4-dinitrophenylsulphenylnitrene (14):¹⁰ the sulphenamide sulphur in ArSNH_2 could be superior in this respect. Thus the diaryl disulphides produced from (24) and (26) could be formed by the route shown in Scheme 5.



An electron-withdrawing substituent in the sulphenamide aromatic ring will adversely affect the availability of a (π -type) sulphur electron pair for attack by an electrophilic sulphenylnitrene. As a consequence other traps may compete successfully for the nitrene. This explanation may account for the intermolecular trapping by alkenes of the sulphenylnitrenes derived from 2-nitro- and 2,4-dinitrobenzenesulphenamides (24) and (13).

The reaction of sulphenamide (13) with allyl aryl sulphides is particularly efficient and, together with

studies of the spin-state of sulphenylnitrene (14), will be reported elsewhere.

EXPERIMENTAL

For details of instrumentation and general experimental directions see ref. 11.

Preparation of Arylsulphenamides (13) and (23)–(26).—2-Nitrobenzenesulphenamide (24) and 2,4-dinitrobenzenesulphenamide (13). A solution of the appropriate sulphenyl chloride (20 mmol) in acetonitrile (50 ml) was added at ambient temperature over 0.5 h to a stirred mixture of ammonia solution (d 0.88; 40 ml) and acetonitrile (30 ml). After a further 0.5 h, saturated sodium chloride solution (100 ml) was added, the organic layer was separated, washed with saturated sodium chloride solution, dried, and evaporated, and the residue was crystallised from ethanol to give 2-nitrobenzenesulphenamide (24) (80%), m.p. 122–125 °C (lit.,¹² 128 °C), and 2,4-dinitrobenzenesulphenamide (13) (85%), m.p. 119–120° (lit.,¹³ 119–120 °C). Benzoylsulphenamide (23) was prepared by the method of Raasch¹⁴ from thiobenzoic acid and hydroxylamine *O*-sulphonic acid, m.p. 81–84 °C (lit.¹⁴ 88.5–90 °C).

4-Nitrobenzenesulphenamide (25). 4-Nitrobenzenethiol (1.55 g) was dissolved in water (100 ml) containing potassium hydroxide (700 mg) and to the filtered solution was added a solution of hydroxylamine *O*-sulphonic acid (2.5 g) and potassium hydroxide (1.5 g) in water (20 ml; cooled to 10 °C) with stirring. The resulting precipitate was separated, washed with water, and dried (1.44 g, 85%), m.p. 105–106 °C (lit.,¹⁵ 101–103 °C).

4-Chlorobenzenesulphenamide (26). To a solution of potassium hydroxide (1.85 g) and 4-chlorobenzenethiol (3.3 g) in water (50 ml) was added, with stirring, a cooled (10 °C) solution of hydroxylamine *O*-sulphonic acid (3.1 g) and potassium hydroxide (1.55 g) in water (10 ml). The resulting mixture was extracted with dichloromethane (2×20 ml) and the combined extracts were washed with water, dried, and evaporated to give the sulphenamide (26) as an unstable oil (1.67 g, 46%), ν_{max} 3380s, 3300s, 1470s, 1390s, 1090s, 1010s and 785 cm^{-1} ; $\delta(\text{CDCl}_3)$ 7.2 (m, $4 \times \text{ArH}$) and 2.65br (s, exch. D_2O , NH_2). This material decomposes to the sulphenimide (27), m.p. 137–138 °C, over a few hours at room temperature but is stable for several weeks at -40 °C. Reaction of (26) (1.0 g) with acetone (2.5 ml) and ammonium chloride (200 mg) by stirring in ethanol (10 ml) for 18 h at ambient temperature gave after filtration, evaporation, and chromatography over basic alumina with light petroleum–ethyl acetate (9:1 v/v), 1,1-dimethyl-*N*-(4-chlorobenzene)sulphenylimine (220 mg, 18%), m.p. 51–53 °C (from light petroleum, b.p. < 40 °C) (lit.,¹⁶ 40–41 °C); $\delta(\text{CDCl}_3)$ 7.4 (AA'BB', $4 \times \text{ArH}$), and 2.10 and 2.05 ($2 \times \text{s}$, $2 \times \text{CH}_3$).

Oxidation of Sulphenamides (13) and (23)–(26) by Lead Tetraacetate in the Absence of Any Trapping Agent.—The sulphenamide (1 mol equiv.) was stirred in dichloromethane (10 ml per g) and powdered lead tetraacetate (1 mol equiv.) was added in portions over 15 min. Evolution of a gas was noted in all cases. Stirring was continued for a further 15 min, then dichloromethane was added and the mixture was filtered and evaporated. Chromatography over basic alumina under the conditions indicated gave the products indicated.

Benzoylsulphenamide (23). Elution with light petroleum

gave a pale yellow solid identified as elemental sulphur (35%) m.p. 116—119°. The other products were intractable.

4-Chlorobenzenesulphenamide (26). Elution with light petroleum—ethyl acetate (9 : 1 v/v) gave bis-(4-chlorophenyl) disulphide (75%), m.p. 68—70 °C (from light petroleum) (lit.,¹⁷ 71.5 °C).

4-Nitrobenzenesulphenamide (25). Elution with light petroleum—ethyl acetate (1 : 1 v/v) gave bis-(4-nitrophenyl) disulphide (33%), m.p. 191 °C (from acetic acid) (lit.,¹⁸ 181 °C).

2-Nitrobenzenesulphenamide (24). Elution with ether gave bis-(2-nitrophenyl) disulphide (49%), m.p. 191—196 °C (from acetic acid) (lit.,¹⁹ 192—195 °C).

2,4-Dinitrobenzenesulphenamide (13). Elution with ethyl acetate—methanol (99 : 1 v/v) gave *N*-2,4-dinitrophenyl-2,4-dinitrobenzenesulphenamide (22) (41%) as a yellow solid, m.p. 231 °C (decomp.) (from nitrobenzene) (Found: C, 38.5; H, 2.0; N, 17.9, C₁₂H₇N₅O₈S requires C, 37.8; H, 1.85; N, 18.4%: figures corrected for 6% occluded nitrobenzene); ν_{\max} . 3 330w, 3 320w, 3 100w, 1 620s, 1 600s, 1 520s, 1 350s, 1 320s, 830s, 740s, and 730s cm⁻¹; δ (DMSO) 9.80 (s, NH), 8.98 and 8.90 (2 d, *J* 3 Hz, H-3 and H-3'), 8.40 and 8.20 (2 dd, *J* 3 and 9 Hz, H-5 and H-5'), and 7.80 and 7.65 (2 d, *J* 9 Hz, H-6 and H-6'); *m/e* 381 (*M*⁺), 199 (base), 183, 153, 150, and 123.

Alternative Synthesis of (22).—2,4-Dinitroaniline (500 mg) was added to a stirred suspension of sodium hydride (150 mg; 50% dispersion in oil) in acetonitrile (80 ml). A solution of 2,4-dinitrobenzenesulphenyl chloride (650 mg) in acetonitrile (30 ml) was added in drops over 0.5 h. After a further 0.5 h hydrochloric acid (2*M*; 5 ml) was added cautiously followed by water (30 ml). The solid obtained was separated and the filtrate was concentrated when more solid was obtained. Chromatography of the combined solids over basic alumina (60 g) and elution with ethyl acetate—methanol (4 : 1 v/v) gave (22) (200 mg, 19%) as yellow crystals (from nitrobenzene), identical with the material obtained above.

Oxidation of 2,4-Dinitrobenzenesulphenamide (13) in the Presence of Alkenes.—**General procedure:** The sulphenamide (13) (1 mol equiv.) and the alkene (5 mol equiv.) (gaseous alkenes were condensed and measured directly into the reaction flask at 0 °C) were dissolved in dichloromethane (7 ml per g of sulphenamide) and powdered lead tetra-acetate (1 mol equiv.) was added portionwise over 15 min with stirring at room temperature (0 °C for gaseous alkenes). After a further 15 min, the mixture was diluted with dichloromethane, filtered, and evaporated and the residue was chromatographed over neutral alumina (Brockmann grade 3). Elution with light petroleum gave unchanged alkene. Further elution with ether gave the aziridines. The following aziridines were obtained in this way: 1-(2,4-dinitrophenylsulphenyl)-2-phenylaziridine (15), from 2,4-dinitrobenzenesulphenamide (13) and styrene as yellow crystals (38%) (from chloroform—light petroleum), m.p. 110—112 °C (Found: C, 52.8; H, 3.5; N, 13.1. C₁₄H₁₁N₃O₄S requires C, 53.0; H, 3.5; N, 13.2%); ν_{\max} . 3 100w, 1 590s, 1 510s, 1 330s, 1 300s, 1 050m, 740s, 730s, and 695s cm⁻¹; δ (CDCl₃) 9.10br (s, aryl H-3), 8.35br (s, aryl H-5 and H-6), 7.35 (m, 5 × phenyl H), 3.15 (dd, *J* 4 and 7 Hz, aziridine H-2), 2.65 (d, *J* 4 Hz, aziridine H-3 *cis* to phenyl), and 2.60 (d, *J* 7 Hz, aziridine H-3 *trans* to phenyl); *m/e* 317 (*M*⁺), 188, 180, and 119; *trans*-1-(2,4-dinitrophenylsulphenyl)-2-methyl-3-phenylaziridine (17), from (13) and (*E*)-1-phenylpropene as orange crystals (64%) (from ethanol), m.p. 115—117 °C (Found:

C, 54.4; H, 4.0; N, 12.7. C₁₅H₁₃N₃O₄S requires C, 54.4; H, 3.95; N, 12.7%); ν_{\max} . 3 100w, 1 595s, 1 520s, 1 330s, 1 300s, and 730s cm⁻¹; δ (CDCl₃) 8.95 (d, 2.5 Hz, aryl H-3), 8.45 (d, *J* 8.5 Hz, aryl H-6), 8.25 (dd, *J* 2.5 and 8.5 Hz, aryl H-5), 7.5br (s, 5 × phenyl H), 3.00 (d, *J* 3.5 Hz, aziridine H-3), 2.85 (dq, *J* 3.5 and 5.5 Hz, aziridine H-2), and 1.60 (d, *J* 5.5 Hz, CH₃); if the crude reaction mixture was chromatographed over basic alumina (instead of neutral alumina) with light petroleum—ethyl acetate (4 : 1 v/v) as eluant, *N*-(2-ethoxy-1-methyl-2-phenylethyl)-2,4-dinitrobenzenesulphenamide (21) was obtained as yellow crystals, m.p. 88—89 °C (from ethyl acetate—light petroleum) (Found: C, 53.9; H, 5.0; N, 11.2. C₁₇H₁₉N₃O₅S requires C, 54.1; H, 5.1; N, 11.1%); ν_{\max} . 3 340w, 3 080w, 1 580s, 1 520s, 1 340s, 1 300s, 1 070s, 910s, 755s, and 700s cm⁻¹; δ (CDCl₃) 9.00 (s, aryl H-3), 8.2 (m, aryl H-5 and H-6), 7.25 (m, 5 × phenyl H), 4.40 (d, *J* 5 Hz, CHOEt), 3.75—2.85 (m, CHMe, NH, and OCH₂), and 1.35 and 1.10 (m, 2 × CH₃), identical to the product obtained by reaction of aziridine (17) with sodium ethoxide—ethanol (see below); 1-(2,4-dinitrophenyl)-2-methyl-2-phenylaziridine (16), from (13) and 2-phenylpropene as an orange glass (61%), ν_{\max} . 3 100w, 1 585s, 1 515s, 1 335s, 1 300s, and 740s cm⁻¹; δ (CDCl₃) 9.00 (d, 1.5 Hz, aryl H-3), 8.30 (m, aryl H-5 and H-6), 7.35 (m, 5 × phenyl H), 2.75br (s, aziridine H-3, *cis* to phenyl), 2.40br (s, aziridine H-3, *trans* to phenyl), and 1.80 (s, CH₃); *trans*-1-(2,4-dinitrophenylsulphenyl)-2,3-dimethylaziridine (18), from (13) and (*E*)-but-2-ene as orange crystals (38%) (from ethanol), m.p. 137—138.5 °C (Found: C, 44.7; H, 4.2; N, 15.65. C₁₀H₁₁N₃O₄S requires C, 44.6; H, 4.1; N, 15.6%); ν_{\max} . 3 110w, 1 590s, 1 510s, 1 340s, 1 300s, 1 080m, 910s, 820s, and 730s cm⁻¹; δ (CDCl₃) 9.10 (d, *J* 2.5 Hz, aryl H-3), 8.50 (d, *J* 8 Hz, aryl H-6); 8.30 (dd, *J* 2.5 and 8 Hz, aryl H-5), 2.40—2.10 (m, 2 × aziridine H), and 1.40 (d, *J* 5.5 Hz, 2 × CH₃); 1-(2,4-dinitrophenylsulphenyl)-2-vinylaziridine (19), from (13) and buta-1,3-diene as orange crystals (58%) (from ethanol), m.p. 113—115 °C (Found: C, 45.1; H, 3.45; N, 15.8. C₁₀H₉N₃O₄S requires C, 44.9; H, 3.4; N, 15.7%); ν_{\max} . 3 100w, 1 590s, 1 515s, 1 380s, 1 300s, 1 050s, 740s, and 730s cm⁻¹; δ (CDCl₃) 9.10 (d, *J* 1.5 Hz, aryl H-3), 8.40 (m, aryl H-5 and H-6), 5.9—5.2 (m, 3 × vinyl H), 2.70 (ddd, *J* 3.7 6.8, and 6.8 Hz, aziridine H-2), 2.40 (d, *J* 3.7 Hz, aziridine H-3, *cis* to vinyl), and 2.38 (d, *J* 6.8 Hz, aziridine H-3 *trans* to vinyl); *cis*-1-(2,4-dinitrophenylsulphenyl)-2-methyl-3-phenylaziridine (20), from (13) and (*Z*)-1-phenylpropene. Using the general procedure a mixture of the *trans*-aziridine (17) and the *cis*-aziridine (20) was obtained. Chromatography over basic alumina with ether as eluant gave the pure *cis*-isomer (20) as yellow crystals (18%) (from ethanol) m.p. 109—110 °C (Found: C, 54.3; H, 3.9; N, 12.7. C₁₅H₁₃N₃O₄S requires C, 54.4; H, 3.95; N, 12.7%); ν_{\max} . 3 120w, 3 090w, 1 595s, 1 515s, 1 330s, 1 300s, 910s, and 730s cm⁻¹; δ (CDCl₃) 9.05 (d, *J* 2.5 Hz, aryl H-3), 8.50 (d, *J* 9 Hz, aryl H-6), 8.35 (dd, *J* 2.5 and 9 Hz, aryl H-5), 7.35br (s, 5 × phenyl H), 3.40 (d, *J* 7.3 Hz, aziridine H-3), 2.65 (dq, *J* 5.5 × 7.3 Hz, aziridine H-2), and 1.20 (d, 5.5 Hz, CH₃); and *trans*-1-(2-nitrophenylsulphenyl)-2-methyl-3-phenylaziridine (28), from 2-nitrobenzenesulphenamide (24) and (*E*)-1-phenylpropene as a yellow oil (58%), δ (CDCl₃) 8.30 (dd, *J* 2.5 and 8 Hz, aryl H-3), 7.65—7.10 (m, 8 × aryl H), 2.90 (d, *J* 4 Hz, aziridine H-3), 2.75 (dq, *J* 4 and 6 Hz, aziridine H-2), and 1.55 (d, *J* 6 Hz, CH₃).

Reaction of *trans*-1-(2,4-Dinitrophenylsulphenyl)-2-methyl-3-phenylaziridine (17) with Sodium Ethoxide—Ethanol.—Sodium (50 mg) was dissolved in ethanol (10 ml) and the

aziridine (17) (200 mg) was added. The mixture was stirred at ambient temperature for 0.5 h then hydrochloric acid (2M; 3 ml) was added and the mixture was extracted with dichloromethane (2 × 5 ml). The organic extracts were dried and evaporated. The n.m.r. spectrum of the residue showed the presence of the sulphenamide (21) (ca. 10%), the remainder of the material being unchanged aziridine (17).

Reaction of Sulphenylaziridines with Sodium Borohydride.—The sulphenylaziridine (0.3 mmol) was stirred with ethanol (1.5 ml) and dichloromethane (2 ml) at room temperature and powdered sodium borohydride (1.2 mmol) was added in portions over 2 min. After having been stirred for a further 10 min the mixture was poured into water (10 ml) and rapidly extracted with dichloromethane (2 × 5 ml). The organic extracts were dried and evaporated and the product was distilled to give the aziridines. Thus 1-(2,4-dinitrophenylsulphenyl)-2-phenylaziridine (15) gave 2-phenylaziridine as oil (45%), b.p. (Kugelrohr) 95–100 °C at 2 mmHg (lit.,²⁰ 94–95 °C at 10 mmHg); ν_{\max} 3 300w, 1 580s, 1 495s, 750s, and 700s cm^{-1} ; $\delta(\text{CDCl}_3)$ 7.25 (s, 5 × ArH), 3.00 (dd, 3.3 and 6 Hz, aziridine H-2), 2.15 (d, *J* 6 Hz, aziridine H-3 *trans* to phenyl), 1.75 (d, *J* 3.3 Hz, aziridine H-3 *cis* to phenyl), and 1.10 (s, NH); and *trans*-1-(2,4-dinitrophenylsulphenyl)-2-methyl-3-phenylaziridine gave *trans*-2-methyl-3-phenylaziridine as an oil (56%), b.p. (Kugelrohr) 110–115 °C at 12 mmHg (lit.,²⁰ 96–97 °C at 10 mmHg); ν_{\max} 3 300w, 1 600m, 1 490s, 1 450s, 840m, and 740s cm^{-1} ; $\delta(\text{CDCl}_3)$ 7.20 (m, 5 × aryl H), 2.65 (d, *J* 2.8 Hz, aziridine H-3), 2.10 (dq, *J* 2.8 and 5.5 Hz, aziridine H-2), 1.35 (d, *J* 5.5 Hz, Me), and 1.20br (s, NH).

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