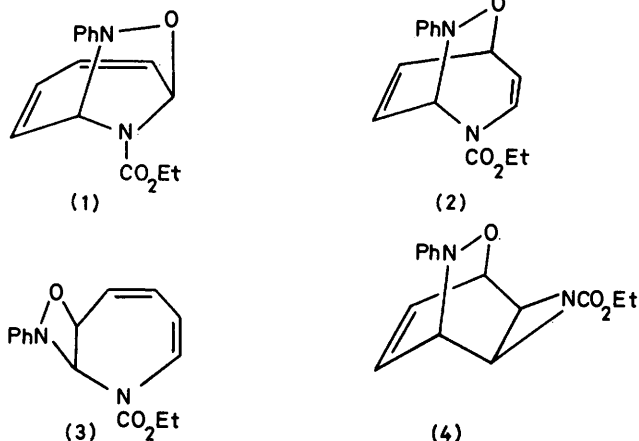


Cycloaddition of Nitrosobenzene to *N*-Ethoxycarbonylazepine

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Nitrosobenzene reacts with *N*-ethoxycarbonylazepine (ethyl 1*H*-azepine-1-carboxylate) to form a cycloadduct. Spectral characteristics indicate a $[6 + 2]\pi$ -type structure. Additional spectroscopic evidence was obtained from a comparison of the hydrogenated derivatives of the azepine adduct and the $[6 + 2]\pi$ cycloadduct of nitrosobenzene and cycloheptatriene. The structure was confirmed as (1) by an X-ray crystallographic study.

In our preliminary report¹ we concluded that the cycloadduct of nitrosobenzene and *N*-ethoxycarbonylazepine (NEA) had the structure (1). This is the product of $[6 + 2]\pi$ cycloaddition. Whilst a $[\pi 6_s + \pi 2_s]$ pericyclic reaction is symmetry forbidden,² an example of a permitted $[\pi 6_s + \pi 2_a]$ reaction has not as yet been proved. The suggestion³ that the cycloadduct of NEA with tetracyanoethylene had a $[6 + 2]\pi$ structure aroused considerable interest but was shown to be erroneous.⁴ The $[6 + 6]\pi$ dimerization of NEA was so unexpected on a symmetry basis that an intermediate was sought and was found.⁵ The reported⁶ $[6 + 2]\pi$ addition of NEA with diethyl azodicarboxylate has now been disproved.⁷ However, the preliminary⁸ report of a $[6 + 2]\pi$ cycloaddition between nitrosobenzene and cycloheptatriene has been confirmed.⁹ This represents the first example of this mode of cycloaddition. The mechanism is unknown. We now report full details of the reaction of NEA with nitrosobenzene.



NEA reacts slowly with nitrosobenzene in benzene at 20 °C. The product, a pale yellow crystalline solid, m.p. 115–116 °C is thermally labile. It was carefully purified by column chromatography using silica. 1 : 1 Cycloaddition was indicated by the mass spectrum of this

¹ W. S. Murphy and J. P. McCarthy, *Chem. Comm.*, 1968, 1155.

² R. B. Woodward and R. Hoffmann, 'The Conservation of Orbital Symmetry,' Verlag Chemie, 1970.

³ K. Hafner, *Angew. Chem.*, 1963, **75**, 1041; *Angew. Chem. Internat. Edn.*, 1964, **3**, 165.

⁴ J. E. Baldwin and R. A. Smith, *J. Amer. Chem. Soc.*, 1965, **87**, 4819; A. S. Kende, P. T. Izzo, and J. E. Lancaster, *ibid.*, 1965, **87**, 5044.

⁵ L. A. Paquette and J. H. Barrett, *J. Amer. Chem. Soc.*, 1966, **88**, 2590; L. A. Paquette, J. H. Barrett, and D. E. Kuhla, *ibid.*, 1969, **91**, 3616.

product, M^+ 272, and by its i.r. spectrum, which had neither OH nor NH stretching frequencies. The most probable structures of the cycloadduct are (1)–(4). Analysis of the i.r. spectrum permitted the qualified rejection of structures (2) and (3). As we have shown elsewhere,⁷ the C=C stretching frequency in the region 1 650–1 625 cm^{-1} is characteristic of a vinylurethane group. The cycloadduct has i.r. absorptions at 1 705 and 1 598 cm^{-1} , with no absorption in the region between. The cycloadduct was hydrogenated to a tetrahydro-derivative which had i.r. absorptions at 1 700 and 1 592 cm^{-1} and no absorptions in the region between. The u.v. difference spectrum between the cycloadduct and its tetrahydro-derivative was λ_{max} (hexane) 260 nm (ϵ 4 142). This spectrum compares favourably with the u.v. difference spectrum between the cycloheptatriene–nitrosobenzene cycloadduct and its tetrahydro-derivative: λ_{max} (EtOH) 250 nm (ϵ 9 882), if a correction¹⁰ is made for the solvent. This spectrum is indicative of a medium-ring *s-cis*-diene chromophore.^{5,8,9,11} However, the vinylurethane chromophore is observed^{4b,7,12} at λ_{max} (EtOH) 240–250 nm. Thus, the u.v. difference spectrum cannot be depended on to differentiate between structures (1)–(3). Structure (4) can now be disregarded. Thus on the basis of the i.r. and u.v. spectra, structures (2)–(4) can be rejected. Henceforth, to facilitate the discussion, the cycloadduct will be provisionally assigned structure (1) and its tetrahydro-derivative, structure (5).

The main fragmentation pathway in the mass spectrum of the cycloadduct is a reverse Diels–Alder type which gives rise to ions, m/e 165 (100%) and 107 (14%). This route is also followed by $[4 + 2]\pi$ cycloadducts.^{7,13} A second pathway followed the same course as did the $[6 + 2]\pi$ nitrosobenzene–cycloheptatriene adduct,⁹ the sequential loss of O, CH, and NCO_2Et leading to the *N*-phenylpyridinium ion, m/e 156 (26%). The third pathway, wherein the adduct gave the peak, m/e 185

⁶ W. S. Murphy and J. P. McCarthy, *Chem. Comm.*, 1970, 1129.

⁷ W. S. Murphy and K. P. Raman, *J.C.S. Perkin I*, 1977, 1824.

⁸ J. Hutton and W. A. Waters, *Chem. Comm.*, 1966, 634.

⁹ P. Burns and W. A. Waters, *J. Chem. Soc. (C)*, 1969, 27.

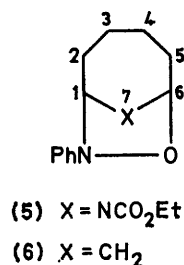
¹⁰ A. I. Scott, 'Ultraviolet Spectra of Natural Products,' Pergamon, Oxford, 1964, p. 57.

¹¹ R. C. Cookson, B. V. Drake, J. Hudec, and A. Morrison, *Chem. Comm.*, 1966, 15; A. L. Johnson and H. E. Simmons, *J. Amer. Chem. Soc.*, 1967, **89**, 3191; A. G. Anastassiou, *ibid.*, 1968, **90**, 1527.

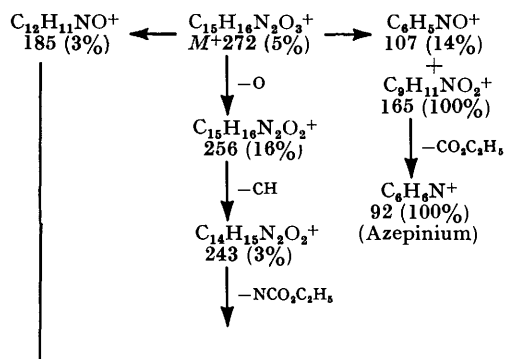
¹² G. Rosencranz, O. Mancera, F. Sondheimer, and C. Djerassi, *J. Org. Chem.*, 1956, **21**, 520.

¹³ T. Sasaki, K. Kanematsu, and K. Hayakawa, *J. Chem. Soc. (C)*, 1971, 2142.

(3%), indicating loss of NCO_2Et provides some evidence for a bridge between C-1 and C-6 (see Scheme). ^1H -azepines, substituted or unsubstituted at positions α to



the nitrogen, do not fragment in this way.¹⁴ $[4 + 2]\pi$ Cycloadducts show little or no tendency to fragment with loss of NCO_2Et :⁷ the $[4 + 2]\pi$ cycloadduct of NEA with 4-phenyl-1,2,4-triazoline-3,5-dione has but a small $M - \text{NCO}_2\text{Et}$ peak, m/e 253 (ca. 0.2%).



SCHEME

In the ^1H n.m.r. spectrum the vinyl protons are observed as a singlet at τ 3.81. This strongly suggests a symmetrical-type structure such as (1). Moreover, it resembles the n.m.r. spectra of related systems.^{5,9,11} The ^1H n.m.r. spectrum of the hydrogenated adduct (5) was compared with those of the hydrogenated $[4 + 2]\pi$ NEA cycloadducts (7)–(9). In structure (5) C-1 and C-6 are each bonded to two electronegative atoms. The protons H-1 and H-6 are observed at τ 4.35 and 4.02 respectively, $\Delta\tau$ 0.33 p.p.m. In structures (7)–(9) only C-1 is bonded to two electronegative atoms and the $\Delta\tau$ values between H-1 and H-5 are much larger (1.41, 1.68, and 1.76 p.p.m.). This spectrum then provides strong evidence for structure (5) and confirms the assignment of structure (1) to the cycloadduct.

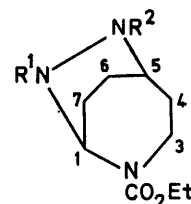
The ^{13}C n.m.r. spectrum of the tetrahydro-derivative of the cycloadduct (5) was investigated. The chemical shifts (Table 1) were assigned by combining multiplicity in the off-centre resonance spectrum with substituent effects.¹⁵ The chemical shift difference between C-6 and C-1, $\Delta\delta_{6-1}$, is 7.86 p.p.m., between C-5 and C-2, $\Delta\delta_{5-2}$, is 2.57, and between C-4 and C-3, $\Delta\delta_{4-3}$, is 0 p.p.m. These assignments were confirmed by comparing the

¹⁴ L. A. Paquette, D. E. Kuhla, J. H. Barrett, and R. J. Haluska, *J. Org. Chem.*, 1969, **34**, 2866.

^{13}C n.m.r. spectrum of the tetrahydro-derivative (6) which has $\Delta\delta_{6-1}$ 11.82, $\Delta\delta_{5-2}$ 2.99, and $\Delta\delta_{4-3}$ 0.13 p.p.m. Also, the two relatively low-field signals for C-1 and C-6 in (5) (see Table 1) could be expected since each

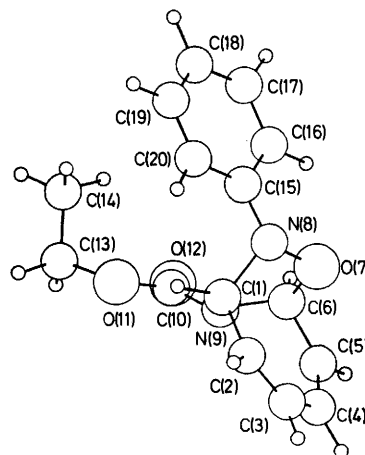
TABLE 1
 Provisional ^{13}C n.m.r. assignments (δ values; tetramethylsilane as standard, solvent CDCl_3)

Carbon	Compound			
	(5)	(6)	(7)	(9)
C-1	79.85	66.08	61.20	61.66
C-2	31.32	32.55		
C-3	23.13	23.78	38.78	38.79
C-4	23.13	23.91	33.78	29.69
C-5	34.89	35.54	47.95	52.30
C-6	87.71	77.90	22.41	20.47
C-7		32.87	23.71	23.97



- (7) $\text{R}^1\text{R}^2 = -\text{OCN}(\text{Ph})\text{CO}-$
 (8) $\text{R}^1\text{R}^2 = -\text{OCC}_6\text{H}_{10}\text{CO}-$
 (9) $\text{R}^1 = \text{R}^2 = \text{CO}_2\text{Et}$

carbon is attached to two electronegative atoms. By comparison compounds (7) and (9) each have only one carbon, C-1, attached to two electronegative atoms. Only one low-field resonance is observed. This conclusion is consistent with the results from the ^1H n.m.r. spectra. We conclude that the NEA-nitrosobenzene cycloadduct has structure (1).



Molecular structure and atom labelling scheme for the cycloadduct (1)

Crystal Structure of the NEA-Nitrosobenzene Cycloadduct.—Since the spectral evidence led to a structure type which has only one precedent in the literature,⁹ a

¹⁵ G. C. Levy and G. L. Nelson, 'Carbon-13 Nuclear Magnetic Resonance for Organic Chemists,' Wiley-Interscience, New York, 1972, p. 47.

structure determination by crystallographic methods was undertaken. Results are given in Tables 2 and 3.

TABLE 2

Atomic co-ordinates for non-hydrogen atoms (fractional parameters $\times 10^4$, with estimated standard deviations in parentheses) and calculated co-ordinates for hydrogen atoms

Atom	x/a	y/b	z/c
C(1)	4 269(5)	5 930(7)	7 110(10)
C(2)	3 526(6)	5 248(9)	7 378(11)
C(3)	2 949(7)	5 773(11)	8 036(14)
C(4)	2 878(7)	7 051(10)	8 753(14)
C(5)	3 423(7)	7 986(11)	9 026(13)
C(6)	4 216(6)	7 869(9)	8 626(10)
O(7)	4 686(5)	6 784(6)	9 651(8)
N(8)	4 944(5)	5 863(7)	8 631(8)
N(9)	4 140(5)	7 378(6)	6 994(8)
C(10)	4 393(5)	8 152(7)	5 863(11)
O(11)	4 473(5)	7 441(6)	4 616(8)
O(12)	4 460(5)	9 324(7)	5 993(8)
C(13)	4 746(8)	8 142(9)	3 375(11)
C(14)	5 627(9)	8 426(16)	3 833(18)
C(15)	5 755(6)	6 190(9)	8 398(11)
C(16)	6 216(7)	7 174(9)	9 267(12)
C(17)	7 008(7)	7 380(13)	9 055(15)
C(18)	7 296(8)	6 611(12)	8 011(15)
C(19)	6 829(8)	5 611(10)	7 159(14)
C(20)	6 064(6)	5 392(9)	7 384(12)
H(1)	4 394(5)	5 485(7)	6 057(10)
H(2)	3 439(6)	4 233(9)	7 008(11)
H(3)	2 456(7)	5 111(11)	8 060(14)
H(4)	2 302(7)	7 274(10)	9 010(14)
H(5)	3 286(7)	8 873(11)	9 598(13)
H(6)	4 507(6)	8 817(9)	8 865(10)
H(131)	4 623(8)	7 563(9)	2 350(11)
H(132)	4 473(8)	9 082(9)	3 223(11)
H(141)	5 790(9)	8 969(16)	2 896(18)
H(142)	5 870(9)	7 513(16)	4 046(18)
H(143)	5 719(9)	9 033(16)	4 919(18)
H(16)	5 973(7)	7 796(9)	10 055(12)
H(17)	7 408(7)	8 101(13)	9 777(15)
H(18)	7 885(8)	6 817(12)	7 807(15)
H(19)	7 054(8)	5 014(10)	6 322(14)
H(20)	5 693(6)	4 594(9)	6 768(12)

TABLE 3

Bond lengths (\AA) and angles ($^\circ$), with estimated standard deviations in parentheses

(a) Distances

C(1)—C(2)	1.514(12)	C(10)—N(9)	1.396(9)
C(1)—N(8)	1.500(10)	C(10)—O(11)	1.323(9)
C(1)—N(9)	1.481(10)	C(10)—O(12)	1.201(9)
C(2)—C(3)	1.358(13)	O(11)—C(13)	1.449(9)
C(3)—C(4)	1.456(14)	C(13)—C(14)	1.478(16)
C(4)—C(5)	1.308(14)	C(15)—C(16)	1.367(12)
C(5)—C(6)	1.479(13)	C(15)—C(20)	1.386(12)
C(6)—O(7)	1.505(10)	C(16)—C(17)	1.421(14)
C(6)—C(9)	1.455(10)	C(17)—C(18)	1.369(16)
O(7)—N(8)	1.423(8)	C(18)—C(19)	1.380(16)
C(15)—N(8)	1.482(10)	C(19)—C(20)	1.380(15)

(b) Angles

C(2)—C(1)—N(8)	109.2(6)	C(1)—N(9)—C(10)	122.6(6)
C(2)—C(1)—N(9)	110.8(6)	C(6)—N(9)—C(10)	120.7(7)
N(8)—C(1)—N(9)	100.1(6)	N(9)—C(10)—O(11)	111.2(6)
C(1)—C(2)—C(3)	126.8(8)	N(9)—C(10)—O(12)	122.4(6)
C(2)—C(3)—C(4)	132.2(10)	O(11)—C(10)—O(12)	126.2(7)
C(3)—C(4)—C(5)	126.4(10)	C(10)—O(11)—C(13)	115.9(6)
C(4)—C(5)—C(6)	123.2(10)	O(11)—C(13)—C(14)	113.3(8)
C(5)—C(6)—O(7)	107.8(7)	N(8)—C(16)—C(15)	121.0(7)
C(5)—C(6)—N(9)	112.7(7)	N(8)—C(15)—C(20)	117.9(7)
O(7)—C(6)—N(9)	102.1(6)	C(16)—C(15)—C(20)	120.8(7)
C(6)—O(7)—N(8)	109.2(5)	C(15)—C(16)—C(17)	118.0(9)
C(1)—N(8)—O(7)	102.1(6)	C(16)—C(17)—C(18)	120.5(11)
C(1)—N(8)—C(15)	113.9(6)	C(17)—C(18)—C(19)	120.8(11)
O(7)—N(8)—C(15)	112.2(6)	C(18)—C(19)—C(20)	118.8(10)
C(1)—N(9)—C(6)	107.4(6)	C(15)—C(20)—C(19)	120.9(9)

The molecular structure shown in the Figure establishes that the cycloadduct has the structure (1). 1,6-Bridging of the azepine nucleus leaves the structure highly strained. This is evident in the bond angles involving C(2)—(4), namely C(2)—C(3)—C(4) 132.2° . The bond angle at C(3), 132° , is most severely strained. The four diene carbons C(2)—(5) are slightly skewed. Both C(3) and C(4) are lifted out of Plane (2) [C(1), C(2), C(3), C(4), C(5)] by 0.042 and -0.041 \AA respectively. The C(3)—C(4) bond length (Table 3) suggests a very small interaction between the two double bonds at C(2) and C(4), in line with their skew conformation.

EXPERIMENTAL

General.—M.p.s were determined with an electrothermal apparatus. I.r. spectra were measured as liquid film or KBr discs on a Perkin-Elmer 257 grating i.r. spectrophotometer. U.v. spectra were recorded for hexane and ethanol solutions on a Perkin-Elmer 402 u.v.—visible spectrophotometer. N.m.r. spectra were recorded for CHCl_3 solutions on a Perkin-Elmer R20 A (60 MHz) spectrometer.

Wet column chromatography was carried out using Merck Kieselgel 60 (70—230 mesh ASTM) and Al_2O_3 (neutral, Brockmann Grade 1). T.l.c. was carried out on plates with Merck silica gel HF254 and visualised under u.v. light and with iodine vapour.

Cycloadduct of Nitrosobenzene and NEA.—Nitrosobenzene (1.92 g) was stirred for 30 min in dry benzene (20 ml) under nitrogen. This solution was added dropwise under nitrogen to NEA (2.97 g) in dry benzene (20 ml). A colour change from intense blue to dark brown took place during 24 h at 20°C . Benzene was removed under reduced pressure and the residue (4.90 g, 100%) was chromatographed through a silica column [ethyl acetate—light petroleum (b.p. $60\text{--}80^\circ\text{C}$)] to give ethyl 8-phenyl-7-oxa-8,9-diazabicyclo[4.2.1]nonane-2,4-diene-9-carboxylate (1) as a light yellow coloured solid (2.45 g, 50%), m.p. $115\text{--}116^\circ\text{C}$ (Found: C, 65.9; H, 6.2; N, 10.2. $\text{C}_{15}\text{H}_{16}\text{N}_2\text{O}_3$ requires C, 66.2; H, 5.9; N, 10.3%); ν_{max} 1705 and 1595 cm^{-1} ; λ_{max} (hexane) 238 (ϵ 6122) and 260 sh nm (ϵ 2585); τ 2.9 (5 H, m, Ph), 3.8 (5 H, s, $4 \times$ vinyl + H-6), 4.5br (1 H, s, H-1), 6.20 (2 H, q, J 8 Hz, OCH_2), and 9.03 (3 H, t, J 8 Hz, CH_3); M^+ 272.

Hydrogenation of the Cycloadduct.—The solution of the adduct (0.5 g) in methanol (25 ml) was shaken with PtO_2 (0.05 g) in a 'Parr' hydrogenator in hydrogen atmosphere (50 lb in^{-2}) for 100 h. Filtration and evaporation of the solvent gave a light yellow oil which when set aside overnight in a refrigerator gave ethyl 8-phenyl-7-oxa-8,9-diazabicyclo[4.2.1]nonane-9-carboxylate, m.p. $50\text{--}51^\circ\text{C}$ (0.507 g, 100%) (Found: C, 65.4; H, 7.6; N, 10.0. $\text{C}_{15}\text{H}_{20}\text{N}_2\text{O}_3$ requires: C, 65.2; H, 7.3; N, 10.1%); ν_{max} 1700 and 1592 cm^{-1} ; λ_{max} (hexane) 239 (ϵ 7152) and 276sh nm (ϵ 1388); τ 2.92 (5 H, m, Ph), 4.03br (1 H, s, H-6), 4.33br (1 H, s, H-1), 6.05 (2 H, q, J 8 Hz, $-\text{OCH}_2$), 8.11br (8 H, s, $4 \times \text{CH}_2$), and 8.98 (3 H, t, J 8 Hz, $-\text{CH}_3$); M^+ 276.

Cycloadduct of Nitrosobenzene and Cycloheptatriene.—A solution of cycloheptatriene (6.0 g) and nitrosobenzene (5.0 g) was set aside in dry ethyl ether (100 ml) for 4 days. Solvent was removed under reduced pressure and the residue extracted with light petroleum (b.p. $40\text{--}60^\circ\text{C}$) ($3 \times 50 \text{ ml}$). The crude product (6 g, 54%) was chromatographed (Al_2O_3 , neutral, eluant: ether—light petroleum) to remove azoxybenzene. The colourless product (1.5 g, 25%) was crystallised from the same solvent; m.p. 106--

107 °C (lit.⁸ 106–107 °C) (Found: C, 78.2; H, 6.5; N, 7.2. Calc. for C₁₃H₁₃NO: C, 78.4; H, 6.6; N, 7.0%); ν_{\max} 1 595 cm⁻¹; λ_{\max} (EtOH) 235 (ϵ 13 432) and 265sh nm (ϵ 6 135); τ 2.98 (5 H, m, Ph), 3.98 (4 H, s, 4 × vinyl), 5.20br (1 H, s, H-6), 5.79br (1 H, s, H-1), and 7.78br (2 H, s, CH₂).

Hydrogenation of the Cycloheptatriene-Nitrosobenzene Adduct.—The adduct (1.0 g) dissolved in methanol (35 ml) was hydrogenated as before for 120 h. Filtration and removal of solvent under reduced pressure gave a viscous oil (1 g, 98%). The crude product was a mixture (t.l.c.) of the cycloadduct and the reduced product. The product was isolated by column chromatography [Al₂O₃, neutral, eluant: ether–light petroleum (b.p. 40–60 °C)] to yield a red oil (0.5 g, 49%) (Found: C, 76.7; H, 8.2; N, 7.0. C₁₃H₁₇NO requires: C, 76.8; H, 8.4; N, 6.9%); ν_{\max} 1 598 cm⁻¹; λ_{\max} (EtOH) 243 (ϵ 12 076) and 280 nm (ϵ 2 199); τ 3.0 (5 H, m, Ph), 5.23br (1 H, d, poorly resolved, H-6), 6.04br (1 H, s, H-1), and 8.31 (10 H, m, 5 × CH₂).

Crystal Data.—Single crystals of the adduct were obtained by slow evaporation of a solution in ethyl acetate–light petroleum (b.p. 60–80 °C) (70:30). C₁₅H₁₆N₂O₃, $M = 272.0$. Monoclinic, $a = 17.01(7)$, $b = 10.18(5)$, $c = 8.54(4)$, $\beta = 104.8(1)^\circ$. $D = 1.30$, $Z = 4$, $D_c = 1.264$, $U = 1 429.74 \text{ \AA}^3$, $F(000) = 576$. Cu- K_α radiation, $\lambda = 1.541.8 \text{ \AA}$; $\mu(\text{Cu-}K_\alpha) = 6.48 \text{ cm}^{-1}$. Space group Cc (C_2^2 No. 9) determined from the systematic absences: hkl , $h + k = 2n + 1$, $h0l$; $l = 2n + 1$ and $0k0$, $k = 2n, +1$. Data were collected by the multiple-film equi-inclination Weissenberg technique for the layers $hk0-7$ and $h0-11$, using Cu- K_α radiation. Intensities were measured by the S.R.C. Microdensitometer Service, Harwell, and 755 unique reflections obtained. Data were corrected for Lorentz and polarisation effects but not for absorption or extinction effects. Data were placed on a common scale by internal correlation and values for atomic scattering factors taken from ref. 16. All calculations were carried out using the SHELX, XANADU, and PLUTO crystallography programmes of Dr. G. Sheldrick and Dr. S. Motherwell (University of Cambridge) on the IBM 370/135 computer at University College, Cork.

Structure Determination.—The structure was solved by

* See Notice to Authors No. 7 in *J.C.S. Perkin I*, 1976, Index issue.

direct methods by use of the tangent refinement facility of the SHELX programme, with the benzene ring as a partial structure. Least-squares analysis involving all non-hydrogen atoms with isotropic temperature factors gave R 0.106 3, with the position of the O(12) atom fixed to define the origin. The position of O(12) was refined by fixing the positions of alternative atoms in the structure in successive stages of the refinement; the map at this stage indicated the position of some of the hydrogen atoms but also showed residual intensities associated with three oxygen, two nitrogen, and the two carbon atoms of the ethoxy-group. Anisotropic temperature factors were calculated for these seven atoms; the positions of the hydrogen atoms were calculated geometrically, and allowed to float on the associated carbon atom assuming C–H 1.08 Å, and with fixed isotropic temperature factor of 0.07 Å. The function minimised in the least-squares refinement was $\Sigma w(|F_o| - |F_c|)^2$ and the weight of each reflection, w , was calculated from the expression: $w = k/(\sigma^2 F_o + g F_o^2)$, where k is a constant approximately equal to unity, which is redetermined after each structure-factor calculation, and g is a small number whose absolute value was refined by fitting $(F_o - F_c)^2$ to $(\sigma^2 F_o + \text{abs}(g) F_o^2)/K$ to yield a g value of 0.014 7 in the present structure. An overall scale factor was also refined. With the final shift to $\sigma < 0.02$, R was 0.066 5 and R' 0.087 5, where $R' = [\Sigma w(|F_o| - |F_c|)^2 / \Sigma w|F_o|^2]^{1/2}$. In the final refinement, nine reflections (with deviations > 3) were suppressed and the residual maximum electron density was 0.33 eÅ⁻³.

Table 2, lists final atomic co-ordinates together with their estimated standard deviations, and Table 3 bond distances and angles. The Figure illustrates the final molecular structure and the atom numbering system used. Final observed and calculated structure factors, relevant mean planes, and thermal parameters are listed in Supplementary Publication No. SUP 22108 (8 pp.).*

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[7/313 Received, 21st February, 1977]

¹⁶ 'International Tables for X-Ray Crystallography,' Kynoch Press, Birmingham, vol. 1, 1969.