m/z 417 (P) 417/416 = 91/9, 399 (P - H_2O) 399/398 = 93/7.

To a solution of 50 mg (0.12 mmol) of **23** in 2 mL of triethylene glycol was added 20 mg of potassium hydroxide. This mixture was heated at 180 °C for 2.75 h under argon. After cooling the mixture was diluted with 30 mL of water and this mixture was made acidic with 1 N HCl. The mixture was extracted throughly with dichloromethane. The combined extract was evaporated to give a residue that was purified by flash chromatography[36] to give 25 mg of **24** as a foam: ^{1}H NMR δ 0.67 and 0.70 (two s, 3 H, 18-CH_3), 0.904, 0.908, 0.915, 0.927, 0.938, 0.968 (3 H, 27-CH_3), 1.01 and 0.985 (two s, 3 H, 19-CH_3), 1.033, 1.036, 1.096, 1.119 (3 H, 21-CH_3), 3.38–3.48 (m, 1 H, 26-C*H*DOH); ^{13}C NMR δ 66.8, 67.1, 67.4, 67.6 (overlapping triplets for C-26); MS, m/z 417 (P) 417/416 = 94/6, 399 (P - H_2O) 399/398 = 95/5. Anal. Calcd for $C_{27}H_{43}DO_3$: C, 77.65; H, 10.86. Found: C, 77.75; H, 10.55.

Acknowledgment. We thank Janis Nelson and Lilia Kurz for NMR spectra.

Products of the Reaction of *N,N'*-Dibenzylethylenediamine and Glyoxal

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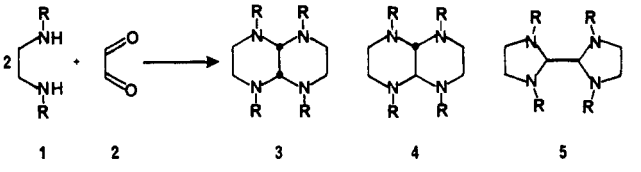
The reaction of *N,N'*-dibenzylethylenediamine with glyoxal in ethanol has been shown to give 1,1',3,3'-tetrabenzyl-2,2'-biimidazolidine (**5c**) and *trans*-1,4,5,8-tetrabenzyl-1,4,5,8-tetraazadecalin (**4c**) in a 60:40 ratio as the initial products. Compound **4c** has been shown to undergo a reversible isomerization to the corresponding cis isomer **3c** in $CDCl_3$ and the $\Delta G°_{333}$ for the isomerization has been determined to be <0.1 kcal/mol. Both **3c** and **4c** show dynamic behavior in their ^{1}H and ^{13}C NMR spectra. In **3c** this dynamic process has a $\Delta G^‡$ of 13.0 kcal/mol and is ascribed to a ring flip process. In **4c** the dynamic process has a $\Delta G^‡$ of 13.3 kcal/mol and is ascribed to slow nitrogen inversion. These results are compared and contrasted to the results obtained in the analogous methyl case.

The reaction of ethylenediamine or an N,N'-disubstituted ethylenediamine with glyoxal can produce three different 2:1 products, the *cis*- and *trans*-1,4,5,8-tetraazadecalins **3** and **4** and the biimidazolidine **5** as summarized in Scheme I.

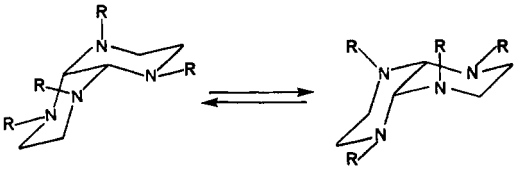
Previous work has established that with ethylenediamine (R = H) the sole product is the *trans*-1,4,5,8-tetraazadecalin **4a**.[1] With *N,N'*-dimethylethylenediamine considerable controversy existed about the structure of the products,[1,2] but it is now clear that the initial product is a mixture of *cis*- and *trans*-1,4,5,8-tetramethyl-1,4,5,8-tetraazadecalins (**3b** and **4b**) rich in the trans isomer.[3] Fuchs et al. were able to synthesize the pure trans isomer **4b** by careful reduction of *trans*-1,4,5,8-tetraethoxycarbonyl-1,4,5,8-tetraazadecalin[3] and observed that **4b** isomerized to the cis isomer **3b**, but because of decomposition of the products were unable to establish if this is a reversible isomerization or a one way process. Katritzky also established that both **3b** and **4b** showed dynamic behavior in their ^{1}H and ^{13}C NMR spectra.[2] The dynamic process in **3b** was ascribed to a ring reversal process between the two lowest energy conformers (see Scheme II). The energy barrier was determined to be 11.6 kcal/mole at 234 °K.

The dynamic process in **4b** was ascribed to *N*-methyl inversion which has a higher than normal energy barrier due to the fact that 1,8 or 4,5 N inversion must occur simultaneously, as summarized in Scheme III, in order to avoid severe peri interactions. This forces the methyl groups to approach very close to each other during the transition state. The barrier was measured to be 9.1 kcal/mol. An unanswered question from Katritzky's work on **4b** (R = CH_3) concerns the relative stability of its achiral (C_i) vs. chiral (C_2) forms of the trans isomer (see Scheme III).

Scheme I. Possible 2:1 Products from the Reaction of an N,N-Disubstituted Ethylenediamine and Glyoxal



Scheme II. Ring Inversion between the Two Lowest Energy Conformers of a *cis*-1,4,5,8-Tetrasubstituted-1,4,5,8-tetraazadecalin 3



Fuchs has reported that the reaction of *N,N'*-dibenzylethylenediamine, **1c**, with glyoxal gives a mixture of two crystalline products, mp 190 °C, one of which is the

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(1) Fuchs, B.; Ellencweig, A. *Recl. Trav. Chim. Pays-Bas* **1979**, *98*, 326.
(2) Ferguson, I. J.; Katritzky, A. R.; Patel, R. *J. Chem. Soc., Perkin Trans. 2* **1976**, 1564.
(3) Fuchs, B.; Weinman, S.; Shmueli, U.; Katritzky, A. R.; Patel, R. *Tetrahedron Lett.* **1981**, *22*, 3541.

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Table I. Chemical Shifts and Coupling Constants Derived from LAOCN Simulation of the ^1H NMR Spectra of the Ethylene Moieties of 3c, 4c, and 5c

		chemical shifts,[a] ppm				coupling constants, Hz				
no.	T, °C	ν_1	ν_2	ν_3	ν_4	$J_{1,2(3,4)}$	$J_{1,3}$	$J_{1,4}$	$J_{2,3}$	$J_{2,4}$
3c[b]	100	2.34	3.09	2.34	3.09	−12.45	3.30	7.27	7.27	3.30
4c[b]	120	2.55	2.75	2.75	2.55	−13.13	3.25	12.40	1.87	3.25
5c[c]	25	2.53	2.98	2.53	2.88	−9.89	6.22	6.03	6.03	6.22

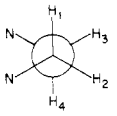
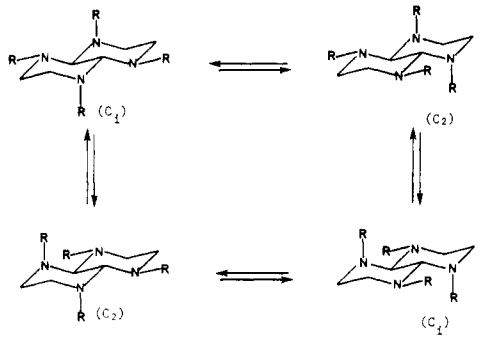
[a] Numbered as below:  [b] Solvent was toluene-d_8. [c] Solvent was $CDCl_3$.

Table II. ^1H NMR Data for Compounds 3c, 4c, and 5c (Excluding Aromatic Protons)

			chemical shifts, ppm			benzyl	ethylene
no.	T, °C	solvent	methine	benzyl CH_2[a]	ring CH_2	J_{AB}, Hz	multiplet type
3c	80	toluene-d_8	3.27	3.26, 4.24	2.23, 3.00	13.2	AA′BB′[b]
	−65	$CDCl_3$	3.36	2.38, 4.61 (ax)		13.5	
				3.83, 4.04 (eq)	not resolved	14.0	ABCD
4c	80	toluene-d_8	3.47	3.79, 4.21	2.42, 2.64	13.4	AA′BB′[b]
	−50	CD_2Cl_2	3.50	3.03, 4.59 (ax)	2.12, 2.62	13.3	AA′BB′
				3.99, 4.57 (eq)	2.45, 3.38	14.8	AA′BB′
5c	80	toluene-d_8	3.42	3.51, 4.29	2.37, 2.90	13.5	AA′BB′
	25	$CDCl_3$	3.49	3.63, 4.43	2.52, 2.98	13.6	AA′BB′[b]

[a] All benzyl methylene spectra appear as AB quartets. [b] See Table I for spin coupling constants.

Scheme III. *N*-Alkyl Inversion in a *trans*-1,4,5,8-Tetrasubstituted-1,4,5,8-tetraazadecalin 4



biimidazolidine **5c**.[1] No attempt to separate these products was reported. It has also been reported that the sole product from the reaction of *N,N′*-diphenylethylenediamine and glyoxal is the biimidazolidine **5d**.[4]

We have now reinvestigated the reaction of *N,N′*-dibenzylethylenediamine and glyoxal. We chose this system because it is very similar to the dimethyl system, yet the products were expected to be crystalline and separable allowing one to study each individually.

Results

When *N,N′*-dibenzylethylenediamine (2 mole equiv) is added to 40% aqueous glyoxal (1 mole equiv) in 95% ethanol at 0 °C, a homogeneous solution results from which colorless crystals start depositing in a few minutes. If these are collected promptly, they prove to be a pure compound, mp 131–132 °C. We have assigned this compound the biimidazolidine structure **5c** based on the following observations. First, it gives the correct elemental analysis for $C_{34}H_{38}N_4$, and it has a 200:1 ratio between the 251 (M/2) and 502 (M) peaks in its EI mass spectrum. Secondly, the ^1H NMR spectrum of the ethylene moiety appears as an AA′BB′ spectrum which can be simulated with coupling constants appropriate for the imidazolidine ring. The chemical shift and coupling constants derived from the LAOCN simulation[5] are summarized in Table I. They are virtually identical with those reported by Fuchs.[1] The compound shows no dynamic effects in either its ^1H or ^{13}C NMR spectra down to −80 °C. The remaining ^1H and ^{13}C NMR data for **5c** are summarized in Tables II and III, respectively.

After the pure **5c** is removed, the reaction mixture then deposits a second crop of crystals which prove to be a mixture of **5c** and another compound. This compound can be obtained in pure form (mp 190–192 °C) by refluxing the mixture containing some added *N,N*-dibenzylethylenediamine for 4 h in 95% ethanol and cooling. Its elemental analysis shows it to be isomeric with **5c**. However, the (M/2)/(M) ratio in its EI mass spectrum is ≈5 indicating that we are dealing with a 1,4,5,8-tetraazadecalin.[1] The ^1H NMR spectrum of this compound shows that it is undergoing a dynamic process at room temperature since both the benzyl protons and ethylene protons are quite broad. The ΔG for this process, measured by the coalescence technique from both ^1H and ^{13}C NMR spectra, is between 13.3 and 13.8 kcal/mol at −5 to −30 °C.[6] Unambiguous assignment of the *trans*-tetraazadecalin structure **4c** was made on the basis of the ^1H NMR spectrum of the ethylene moiety. At high temperature (120 °C) where N inversion is rapid, the ethylene fragment of **4c** should give rise to an AA′BB′ spectrum with appropriate gauche and anti coupling constants, as indeed it does. The coupling constants and chemical shifts derived from a LAOCN simulation are summarized in Table I.

This assignment is supported by the low-temperature ^1H and ^{13}C NMR spectra of **4c**. In the ^1H NMR spectrum there are two AA′BB′ patterns for the two different ethylene moieties expected in the C_2 form of **4c**. We were unable to observe the ABCD pattern expected for the

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(5) Castellano, S.; Bothner-By, A.; *J. Chem. Phys.* **1964**, *41*, 3863–3869. The adaptation of this program known as ITRCL written for the Nicolet 1280 computer was used for the spectral analysis.

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Table III. ^{13}C NMR Chemical Shifts of 3c, 4c, and 5c (50.3 MHz, Excluding Protonated Aromatic Carbons)

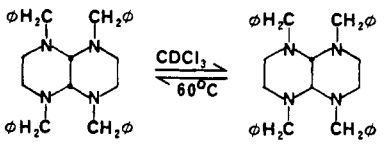
			chemical shift, ppm (Me_4Si)			
no.	solvent	T, °C	bridge CH	ring CH_2	benzyl CH_2	aromatic C-1
3c	toluene-d_8	80	71.88	43.38	55.12	137.99
	$CDCl_3$	−50	71.79	41.52, 45.09	54.36, 56.30	138.50, 139.35
4c	toluene-d_8	80	72.32	43.96	49.76	135.53
	CD_2Cl_2	−60	73.23	40.69, 49.58	45.96, 55.59	138.68, 140.85
5c	$CDCl_3$	25	88.59	50.41	60.74	140.40

ethylene moiety in the C_i form of **4c**. The ^{13}C NMR spectrum of **4c** shows a single five-line pattern in the aliphatic region. Thus, based on both the ^1H and ^{13}C NMR data it appears that **4c** exists in solution largely as the C_2 conformer. The remaining ^1H and ^{13}C NMR data for **4c** are summarized in Tables II and III, respectively.

When the solution of **4c** was heated in the course of the variable temperature ^1H NMR experiments, a second set of signals was observed to grow in. They proved to belong to a third isomer of **4c** and **5c** which can be isolated relatively pure by heating a solution of pure **4c** at 60 °C in $CDCl_3$ for 18 h, removing the solvent and triturating with pentane. This third compound is very soluble in pentane while **4c** is virtually insoluble. This compound was assigned the *cis*-tetraazadecalin structure **3c** on the basis of the high-temperature ^1H NMR spectrum of its ethylene moiety. When ring reversal is rapid (100 °C), the ethylene moiety of **3c** appears as an AA′BB′ spectrum with averaged coupling. The coupling constants and chemical shifts derived from a LAOCN simulation are summarized in Table I. Compound **3c** also shows, as expected, dynamic behavior in its ^1H and ^{13}C NMR spectra. We have measured the $\Delta G^{\ddagger}$ for this process to be 13.0 kcal/mol from the coalescence temperatures of the benzyl protons and benzyl carbons. Structure **3c** is supported by the low-temperature ^1H and ^{13}C NMR spectra: the ethylene moiety appears as an ABCD pattern, there are two different AB patterns for the equatorial and axial benzyl groups in the ^1H NMR spectrum, and the ^{13}C NMR spectrum shows a doubling of all resonances in the aliphatic region except for the bridgehead carbons. Again, the ^1H and ^{13}C NMR data for **3c** are summarized in Tables II and III, respectively.

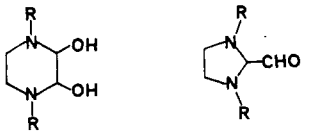
One of the unanswered questions from Katritzky's work on the *cis*- and *trans*-tetramethyltetraazadecalins (**3b** and **4b**) was whether the conversion of the trans to the cis isomer represented an equilibrium or a one way process. Since we had pure samples of each of the corresponding benzyl isomers, we have been able to establish that in this series a palpable equilibrium exists since pure samples of either **3c** or **4c** reach the same composition of 51% **4c** and 49% **3c** when heated at 60 °C in $CDCl_3$ for approximately 18 h (Scheme IV).

Scheme IV. Equilibration between *cis*- and *trans*-1,4,5,8-Tetrabenzyl-1,4,5,8-tetraazadecalins 3c and 4c



Discussion

We felt when we started this project that our results should be very similar to those of Katritzky[2,3] for the methyl case since methyl and benzyl groups have very similar conformational free energy values (1.70 kcal/mol for methyl[7] and 1.81 kcal/mol for benzyl[8]). However, it is now clear that there are some substantial differences between the two systems. First, we (and Fuchs)[1] observed the biimidazolide product as one of the initial products in the benzyl case while such a species was not observed in the methyl case. This is probably related to the relative rates with which the intermediate 2,3-dihydroxypiperazines **6b** and **6c** either react with an additional mole of diamine leading to the decalin product, or undergo rearrangement to the 2-imidazolidine carboxaldehydes **7b** and **7c** leading to the biimidazolidine product.[9]



6 7

It is striking that no *cis*-1,4,5,8-tetrabenzyl-1,4,5,8-tetraazadecalin, **3c**, is observed in the initial product even though it is of essentially equal energy with the trans isomer **4c** as shown by the equilibration experiments. This must mean that, whatever the mechanism by which the tetraazadecalin product is formed, it greatly favors the trans form kinetically.

Katritzky measured a value of 9.1 kcal/mol for the *N*-methyl inversion in **4b** while we have determined the analogous barrier in **4c** to be 13.3–13.8 kcal/mol. This large difference is hard to explain simply on the basis of the A values for methyl and benzyl group. The barrier measured for ring flip in **3c**, 13.0 kcal/mol, is reasonably close to that measured for **3b** (11.6 kcal/mol).

The equilibration experiments clearly show that the **3c** → **4c** isomerization and the analogous methyl compounds **3b** and **4b** are reversible. The fact that **3c** and **4c** turn out to be essentially equal in energy must be fortuitous. The ratio of the C_2 to C_i forms of **4c** is surprising and may be difficult to explain.

Experimental Section

NMR Spectra were recorded on Nicolet WB-200 and General Electric GN-300 Spectrometers and both ^1H and ^{13}C spectra are reported on the δ scale. Mass spectra were recorded on a Hewlett-Packard 5895B GC/MS system operating at 70 eV for the electron impact spectra and using methane as the reagent gas for the chemical ionization spectra. Microanalyses were carried out by Galbraith Laboratories, Inc.

1,1′,3,3′-Tetrabenzyl-2,2′-biimidazolidine (5c). A solution of 40% aqueous glyoxal (1.45 g, 10 mmoles) in 50 mL of ethanol is placed in a 250-mL Erlenmeyer flask. This solution is cooled to 0 °C (salt–ice bath) and stirred while a solution of *N*,*N*′-dibenzylethylenediamine (4.8 g, 20 mmol) is added dropwise over 10 min. Approximately 5 min after the addition is complete, crystals start depositing on the sides of the reaction flask. If these

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(9) Willer, R. L.; Moore, D. W.; Vanderah, D. J.; Lowe-Ma, C. K. *J. Org. Chem.*, following paper in this issue.

are collected after approximately 10 additional min, they prove to be fairly pure **5c**. However, if the reaction is allowed to run longer, the product becomes a mixture of **5c** and **4c** in a ratio of ≈40:60.

Anal. Calcd for $C_{34}H_{38}N_4$: C, 81.23; H, 7.62; N, 11.15. Found: C, 80.92; H, 7.75; N, 11.15.

***trans*-1,4,5,8-Tetrabenzyl-1,4,5,8-tetraazadecalin (4c).** A solution of 40% aqueous glyoxal (1.45 g, 10 mmol) in 50 mL of ethanol is stirred while a solution of *N,N*-dibenzylethylenediamine (7.2 g, 30 mmol) in 50 mL of ethanol is added dropwise. After the addition is complete the mixture is refluxed for 3 h. The mixture is cooled, the product collected, and the product is then washed with ethanol. After drying, the product weighs 2.09 g (4.1 mmol, 41%) and melts at 190–192 °C.

Anal. Calcd for $C_{34}H_{38}N_4$: C, 81.23; H, 7.62; N, 11.15. Found: C, 81.11; H, 7.66; N, 11.13.

***cis*-1,4,5,8-Tetrabenzyl-1,4,5,8-tetraazadecalin (3c).** A solution of 1.0 g of pure **4c** in 5.0 mL of $CDCl_3$ is heated at 60 °C under nitrogen for 18 h. The solvent is removed at reduced pressure and the resulting semisolid triturated with 20 mL of pentane. The pentane is removed at reduced pressure to give 0.45 g of **3c** as a gummy oil.

Acknowledgment. This work was funded under the Naval Weapons Center Independent Research Task and the NAVSEA Explosive Research and Development Program Task SR02403. The authors are indebted to Ralph Hurd of General Electric NMR for the 300-MHz ^1H NMR spectra of **3c** and **4c**.

Registry No. **3c**, 96482-22-3; **4c**, 96444-73-4; **5c**, 96444-74-5; $PhCH_2NH(CH_2)_2NHCH_2Ph$, 140-28-3; glyoxal, 107-22-2.

New Chemistry from the Reaction of N,N′-Disubstituted Ethylenediamines with Glyoxal: Synthesis of 2-Imidazolidinecarboxaldehydes and 1,4,6,9-Tetraalkyl-1,4,6,9-tetraaza-5,10-dioxaperhydroanthracenes

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The reaction of *N,N′*-di-*tert*-butylethylenediamine with glyoxal in water gives initially *trans*-2,3-dihydroxy-1,4-di-*tert*-butylpiperazine, **6f**, which rearranges thermally to 1,3-di-*tert*-butyl-2-imidazolidinecarboxaldehyde, **8f**, and then to 1,4-di-*tert*-butyl-2-ketopiperazine, **5f**. The reaction of *N,N′*-diisopropylethylenediamine with glyoxal in water produces 1,4-diisoproyl-2-ketopiperazine, **5e**, as the only isolable product. The reaction of a series of N,N′-dialkyl-substituted ethylenediamines with glyoxal in ethanol at low temperature has been found to give a series of *cis-trans-cis*-1,4,6,9-tetraalkyl-1,4,6,9-tetraaza-5,10-dioxaperhydroanthracenes, **9b,c,e**, as minor products. The crystal structure of **9e** was determined confirming the stereochemistry of the ring junctures. *N,N′*-Diphenylethylenediamine reacts with glyoxal to give 1,3-diphenyl-2-imidazolidinecarboxaldehyde, **8d**. **8d** shows no tendency to rearrange to **5d**. A modified reaction scheme for the reaction of N,N′-disubstituted ethylenediamines with glyoxal is presented which accounts for the formation of these new types of products.

The reactions of ethylenediamine and N,N′-disubstituted ethylenediamines, **1a–d**, with glyoxal have been the subject of several investigations.[1-9] The reactions have been found to yield four different types of products, namely *trans*-1,4,5,8-tetraazadecalins, **2**, *cis*-1,4,5,8-tetraazadecalins, **3**, 2,2′-biimidazolidines, **4**, and lactams **5** (see Scheme III for structures). The product(s) obtained has been found to depend both on the amine substituent and the reaction conditions. When R is hydrogen, the product is the *trans*-tetraazadecalin **2a**.[1,2] When R is methyl, the product is a mixture of the *trans*- and *cis*-tetraazadecalins **2b** and **3b** when the reaction is run under mild conditions[3,4] and the lactam **5b** when the reaction is run under more vigorous conditions.[2] When R is benzyl, the product is a mixture of the *trans*-tetraazadecalin **2c** and biimidazolidine **4c**.[5] The *trans*-tetraazadecalin **2c** is in equilibrium with the cis isomer **3c** at elevated temperatures in chloroform. When R is phenyl, the product is the 2,2′-biimidazolidine **4d**.[6] Fuchs has proposed a reaction scheme to account for these products.[2]

As an outgrowth of our interest in diamine–glyoxal chemistry,[5,10] we have studied the reaction of *N,N′*-diisopropylethylenediamine, **1e**, and *N,N′*-di-*tert*-butylethylenediamine, **1f**, with glyoxal and reexamined the reaction of **1b**, **1c**, and **1d** with glyoxal under different conditions. In this paper we report our experimental results and show how they fit into an expanded reaction scheme.

Results

We first examined the reaction of *N,N′*-di-*tert*-butylethylenediamine (**1f**) with glyoxal. The addition of 1 mol equiv of **1f** to a well-stirred aqueous solution of glyoxal

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‡ 1982 American Society for Engineering Education Summer Faculty Research Participant at the Naval Weapons Center.

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