had mp 290 °C (lit.[28] mp 295 °C).

2-Aminododecanoic acid (8c): 4.30 g, 100%; mp 260 °C dec. (lit.[28] mp 263 °C dec); IR (Nujol) 3300, 2100, 1610, 1585 cm^{-1}; ^1H NMR (DMSO-d_6 + DCl) δ 0.50–1.90 (m, 21 H), 3.90–3.96 (m, 1 H).

Phenylglycine (8d): 2.75 g, 91%; mp 263–265 °C dec (lit.[28] mp 256 °C, subl); IR (Nujol) 3100, 2100, 1660, 1630, 1590 cm^{-1}; ^1H NMR (DMSO-d_6 + DCl) δ 5.30 (s, 1 H), 7.55 (s, 5 H).

2-(2-Methylphenyl)glycine (8e): 2.97 g, 90%; mp 234–238 °C dec; IR (Nujol) 3200, 2080, 1630, 1570 cm^{-1}; ^1H NMR (DMSO-d_6 + DCl) δ 2.55 (s, 3 H), 5.52 (s, 1 H), 7.30–7.60 (m, 4 H). Anal. Calcd for $C_9H_{11}NO_2$: C, 65.43; H, 6.72; N, 8.48. Found: C, 65.36; H, 6.81; N, 8.51.

2-(3-Methoxyphenyl)glycine (8f): 3.30 g, 91%; mp 215 °C dec (lit.[29] mp 220–222 °C dec); IR (Nujol) 3200, 2060, 1605, 1585 cm^{-1}; ^1H NMR (DMSO-d_6 + DCl) δ 3.90 (s, 3 H), 5.18 (s, 1 H), 7.00–7.55 (m, 4 H).

Phenylalanine hydrochloride (8j): 4.01 g, 100%; IR (Nujol) 3230, 2200, 1730, 1595 cm^{-1}; ^1H NMR (DMSO-d_6) δ 3.05–3.20 (m, 2 H), 3.80–4.15 (m, 1 H), 7.10–7.25 (m, 5 H). Free **8j** was obtained with a basic resin and had mp 280 °C (lit.[28] mp 284–288 °C).

2-Piperidinecarboxylic acid hydrochloride (11): 3.21 g, 97%; mp 255 °C (lit.[30] mp 259–261 °C); IR (Nujol) 3480, 2110, 1730, 1585 cm^{-1}; ^1H NMR (DMSO-d_6) δ 1.10–2.60 (m, 6 H), 2.90–3.50 (m, 2 H), 4.15–4.45 (m, 1 H).

The alkylation products of CF_3CONH_2 (1) with 2-bromo carboxylic esters **6g–i** were not isolated, but the crude of reaction, after filtration and evaporation of the solvent, was directly hydrolized following the general procedure described above. The yield and physical and spectroscopic data are the following:

2-(4-Fluorophenyl)glycine (8g): 2.44 g, 72% (yield based on the starting 2-bromo ester **6g**); mp 270–272 °C dec (lit.[31] mp 271–273 °C); IR (Nujol) 3080, 2100, 1620, 1580, 1115 cm^{-1}; ^1H NMR (DMSO-d_6 + DCl) δ 5.28 (s, 1 H), 7.25–7.80 (m, 4 H).

2-(4-Chlorophenyl)glycine (8h): 2.46 g, 70% (overall yield): mp 272–274 °C, dec (lit.[31] mp 270–272 °C dec); IR (Nujol) 3060, 2100, 1620, 1580 cm^{-1}; ^1H NMR (DMSO-d_6 + DCl) δ 5.33 (s, 1 H), 7.45–7.66 (m, 4 H).

2-(4-Bromophenyl)glycine (8i): 3.11 g, 68% (overall yield); mp 262–264 °C (lit.[32] mp 265 °C subl); IR (Nujol) 3080, 2100, 1630, 1590 cm^{-1}; ^1H NMR (DMSO-d_6 + DCl) δ 5.25 (s, 1 H), 7.40–7.80 (m, 4 H).

Extractability of Carbonate Ion in the Organic Phase. Anhydrous potassium carbonate (6.90 g, 50 mmol) and CH_3CN (50 mL) were magnetically stirred at 80 °C for 20 min both in the absence and presence of benzyltriethylammonium chloride (TEBA, 1.14 g, 5 mmol). The stirring was stopped, and aliquots (5 mL) of the organic phase were withdrawn and titrated with 0.01 N HCl (potentiometric titration). No basic species were detected in either case.

Extractability of Trifluoroacetamide Anion as Potassium or Quaternary Ammonium Salt in the Organic Phase. An acetonitrile solution (50 mL) of CF_3CONH_2 (1) (5.65 g, 50 mmol) was stirred over anhydrous K_2CO_3 (6.90 g, 50 mmol) at 80 °C for 20 min. The acid titration of aliquots (5 mL) of organic phase showed the presence of basic species (0.06 mol/mol of starting 1). When the above run was performed in the presence of TEBA (1.14 g, 5 mmol) the basic species reached 0.10 mol/mol of 1.

^{19}F NMR Measurements. The ^{19}F NMR spectrum of a CD_3CN solution of trifluoroacetamide (1) showed a singlet at ca. −65 ppm. The ^{19}F NMR spectrum of an equimolar solution of 1 and preformed CF_3CONHK[2] in CD_3CN showed two singlets at ca. −65 and −64 ppm, the latter can be assigned to fluorine of the CF_3CONH^-. When a CD_3CN (5 mL) solution of 1 (0.57 g, 5 mmol) was stirred over anhydrous K_2CO_3 (0.69 g, 5 mmol) for 20 min at 80 °C, ^{19}F NMR analysis showed, together with the signal of 1, the presence of the singlet at ca. −64 ppm. From the integrals of the two signals 0.06 mol of CF_3CONH^-/mol of 1 was evaluated, in agreement with the results obtained by potentiometric titrations. In a similar run, but carried out in the presence of TEBA (0.11 g, 0.5 mmol), the amount of CF_3CONH^- reached 0.10 mol/mol of 1, as previously found by acid titrations.

Acknowledgment. The present work has been supported by a grant from the Progetto Finalizzato Chimica Fine II of the CNR Roma.

Registry No. 1, 354-38-1; **6a**, 105-36-2; **6b**, 41978-69-2; **6c**, 129592-86-5; **6d**, 2216-90-2; **6e**, 129592-87-6; **6f**, 86215-57-8; **6g**, 129592-88-7; **6h**, 129592-89-8; **6i**, 129592-90-1; **6j**, 129592-91-2; **6k**, 70288-66-3; **7a**, 367-62-4; **7b**, 26629-29-8; **7c**, 129592-92-3; **7d**, 129592-93-4; **7e**, 129592-94-5; **7f**, 129592-95-6; **7j**, 16417-60-0; **7k**, 129592-97-8; **8a**·HCl, 6000-43-7; **8b**·HCl, 25616-13-1; **8b** (free base), 302-72-7; **8c**, 35237-37-7; **8d**, 2835-06-5; **8e**, 129592-98-9; **8f**, 7314-43-4; **8g**, 7292-73-1; **8h**, 7292-70-8; **8i**, 129592-99-0; **8j**·HCl, 27172-85-6; **8j** (free base), 150-30-1; **9**, 4192-77-2; **10**, 129592-96-7; 11·HCl, 5107-10-8.

(28) *Dictionary of Organic Compounds*; Chapman and Hall: London, 1982.
(29) Neims, A. H.; De Luca, D. C.; Hellerman, L. *Biochemistry* **1966**, *5*, 203.
(30) Schniepp, L. E.; Marvel, C. S. *J. Am. Chem. Soc.* **1935**, *57*, 1557.
(31) Compere, E. L.; Weinstein, D. A. *Synthesis* **1977**, 852.
(32) Wislicenus, W.; Elvert, H. *Chem. Ber.* **1908**, *41*, 4121.

A New and Efficient Method for the Resolution of 2,2'-Dihydroxy-1,1'-binaphthyl

Bao-qing Gong, Wan-yi Chen, and Bing-fang Hu*

Department of Applied Chemistry, Beijing Agricultural University, Beijing, People's Republic of China

Received February 9, 1990

In the past few years, much attention has been focused on the studies of the chirality recognition properties of chiral crown ether hosts containing the binaphthol unit.[1] Also, the application of binaphthyl-containing chiral catalysts or reagents in asymmetric synthesis has proven fruitful.[2] Consequently, the preparation of optically active binaphthol is of current interest. Its conventional large-scale preparation relies on the optical resolution of its cyclic phosphoric ester using cinchonine as a resolving agent.[3] By this method, the overall resolved yield is only moderate (41% for (+)-*R*-1; 52% for (−)-*S*-1), and the enantiomeric purity of the product is even less satisfactory (96.6% for (+)-*R*-1).[1] Cinchonine is expensive and often recovered in contaminated from. Recently, a method for preparing (−)-*S*-1 by the coupling of the *S*-(+)-amphetamine-copper(II) complex of β-naphthol has been reported.[4] It is attractive owing to its simplicity, but the amine is expensive and is needed in large quantity (1:8 mol ratio). An efficient method of optical resolution by enantioselective complex formation using specially prepared tartaric amide has also been described recently.[5] Jacques's[6] and Truesdale's[7] work improved Cram's procedure by preparing a purer BNP acid and resolving it with cinchonine. The enantiomeric acids were methylated to the esters, which were reduced by Red-Al (Aldrich). The enantiomeric purity of each resulting binaphthol was higher, but the procedure is composed of a number of preparative steps, each entailing separations and purifications. This resulted in a lower overall yield (34% for (*R*)-(+)-2,2'-dihydroxy-1,1'-binaphthyl and 39% for the *S*-(−) enantiomer) than Cram's procedure.

We now report a new and more efficient method for this enantiomeric resolution via the formation of the phos-

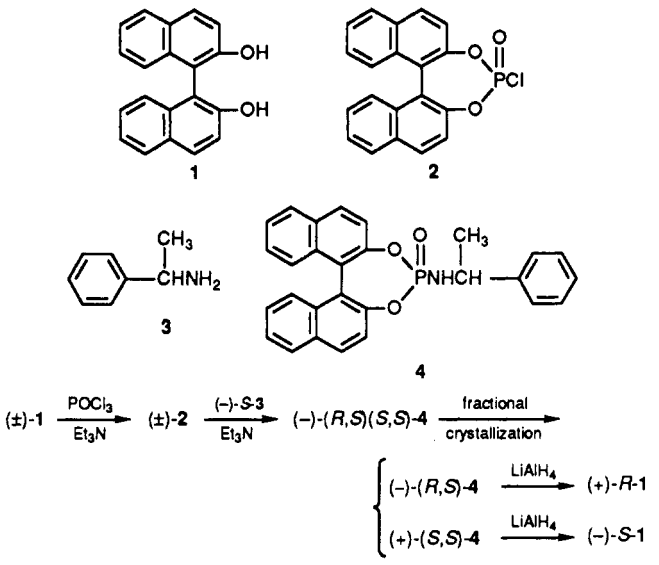
(1) Kyba, E. P.; Gokel, G. W.; de Jong, F.; Koga, K.; Sousa, L. R.; Siegel, M. G.; Kaplan, L. G.; Dotsevi, G.; Sogah, Y.; Cram, D. J. *J. Org. Chem.* **1977**, *42*, 4173.
(2) Miyashita, A.; Noyori, R. *J. Am. Chem. Soc.* **1980**, *102*, 7932.
(3) Jacques, J.; Fouguoy, C. *Tetrahedron Lett.* **1971**, 4617.
(4) Brussee, J.; Groenendijk, J. L. G.; te Koppele, J. M.; Jansen, A. C. A. *Tetrahedron* **1985**, *41*, 3313.
(5) Toda, F.; Tanaka, K. *J. Org. Chem.* **1988**, *53*, 3607.
(6) Jacques, J.; Fouguey, C. *Org. Synth.* **1988**, *67*, 1–12.
(7) Truesdale, L. K. *Org. Synth.* **1988**, *67*, 13–19.

0022-3263/91/1956-0423$02.50/0

Table I. Resolution of 2,2′-Dihydroxy-1,1′-binaphthyl

resolving agent	4	mp, °C	$[\alpha]^{20}_D$, deg, c = 1.0, $CHCl_3$	yield, %	1	mp, °C	$[\alpha]^{20}_D$, deg, c = 1.0, THF	enantiomeric purity, %	yield, %	overall yield, %
(−)-*S*-**3**	(+)-(*S*,*S*)-**4A**	251–3	+388	88	(−)-*S*-**1**	206–7	−35.2	100	92	70.9
	(−)-(*R*,*S*)-**4B**	267–9	−379	77	(+)-*R*-**1**	206–7	+34.9		90	60.7
(+)-*R*-**3**	(−)-(*R*,*R*)-**4C**	251–3	−388	85	(+)-*R*-**1**	206–7	+35.2	100	92	68.5
	(+)-(*S*,*R*)-**4D**	267–9	+379	70	(−)-*S*-**1**	206–7	−34.8		90	55.2

Scheme I



phoramidate with optically active phenethylamine, a most readily accessible and widely used basic resolving agent. Compared to the conventional methods, a significant enhancement of overall yield (69% for (+)-*R*-**1**; 71% for (−)-*S*-**1**) and enantiomeric purity (100% for both enantiomers) can be achieved. The resolving agent can be recovered in 80% yield with its original enantiomeric purity. The main advantage of this procedure over that described in ref 6 and 7 lies in the unusual difference in solubility behavior between the two diastereomers **4A** (*S*,*S*) and **4B** (*R*,*S*), or **4C** (*R*,*R*) and **4D** (*S*,*R*). This enables an efficient resolution in one recrystallization from ethanol, affording products in nearly 100% enantiomeric purity (observed by ^{31}P NMR). In contrast, the method of ref 6 produces the crystalline (±)-BNP cinchonine salt from a methanol–water mixture as a 91:9 mixture of the diastereomeric salts. Our experimental findings show that our econonical method enables large quantities of both enantiomeric binaphthols of high enantiomeric purity to be made. The general scheme for this resolution pathway is shown in Scheme I.

Using (+)-*R*-**3**, the steps and workup are the same as above and these results are summarized in Table I.

Experimental Section

The values of specific rotation were measured on a polartronic D. Schmide + Haensch polarimeter. The melting points were determined on a Yanaco apparatus. ^1H NMR data were recorded with a JEOL FX-900 spectrometer, using TMS as internal standard, ^{31}P NMR data were recorded using 85% H_3PO_4 as external standard. Mass spectra were taken on a FINNIGAN MAT 4510 mass spectrometer.

Preparation of (*R*,*S*)-1,1′-Binaphthyl-2,2′-diyl-*N*-(α-(*S*)-methylbenzyl)phosphoramidate (4A,B). (±)-1,1′-Binaphthyl-2,2′-diylphosphoryl chloride was prepared according to the known procedure.[1,3] A solution of (−)-*S*-**3** (2.5 g, 20 mmol) and triethylamine (2.3 g, 23 mmol) in dichloromethane (15 mL) was added dropwise with constant stirring into crude **2** (from 20 mmol of racemic binaphthol), cooled in an ice–salt bath. After the addition (0.5 h), the mixture was stirred at room temperature for 36 h. The reaction mixture was washed with 4% aqueous HCl and then with saturated brine and was dried over anhydrous $MgSO_4$. After the removal of solvent under reduced pressure, crystalline solid (8.8 g) was obtained. The ^{31}P NMR data indicated that the product was the expected **4A** and **4B** (12.17 ppm, 12.68 ppm, $CHCl_3$) with slight contamination by the corresponding phosphoric ester (4.60 ppm, $CHCl_3$). The crude product was then chromatographed by the VLC method[8] (silica gel, 300 mesh, 1:10) with elution by anhydrous ether. A pure mixture of **4A** and **4B** was obtained (7.9 g, 87.6% yield): mp 215–219 °C; ^{31}P NMR 12.17 and 12.68 ppm; $[\alpha]^{20}_D$ +31.0° (*c* =1.0, $CHCl_3$); MS (M^+) 451.

Separation of 4A and 4B. Pure **4** (7.9 g) was dissolved in absolute ethanol (120 mL) under reflux. After 48 h at room temperature, the solution was filtered, giving **4A** as crystalline plates (3.48 g, 88% yield): mp 251–253 °C; $[\alpha]^{20}_D$ +388° (*c* = 0.5, $CHCl_3$); repeated recrystallization gave the product with the same melting point and $[\alpha]^{20}_D$; ^{31}P NMR 12.17 ppm ($CDCl_3$) (single signal); ^1H NMR ($CDCl_3$) δ 1.58 (d, CH_3, 3 H), 4.60 (m, CH, 1 H), 5.52 (t, NH, 1 H), 7.60–7.32 (m, Ar-H, 13 H), 8.20–8.01 (m, Ar-H, 4 H). Anal. Calcd for $C_{28}H_{22}O_3NP$: C, 74.48; H, 4.92; N, 3.10. Found: C, 74.47; H, 4.97; N, 2.90.

The filtrate was concentrated under reduced pressure to half its volume and then refluxed to dissolve the solid. After 36 h at room temperature, it afforded crystalline needles **4B**, which gave pure **4B** after recrystallization (3.13 g, 77% yield): mp 267–269 °C; $[\alpha]^{20}_D$ −379°; ^{31}P NMR 12.65 ppm ($CHCl_3$) (single signal); ^1H NMR ($CDCl_3$) 1.50 (d, CH_3, 3 H), 4.58 (m, CH, 1 H), 5.42 (t, NH, 1 H), 7.60–6.70 (m, Ar-H, 13 H) 8.20–7.92 (m, Ar-H, 4 H). Anal. Calcd for $C_{28}H_{22}O_3NP$: C, 74.48; H, 4.92; N, 3.10. Found: C, 74.38; H, 4.97; N, 2.92.

Using (+)-*R*-**3**, the corresponding phosphoramidate was likewise obtained in 87.6% yields: mp 215–219 °C; ^{31}P NMR 12.17 and 12.68 ppm ($CHCl_3$); $[\alpha]^{20}_D$ −31.0° (*c* = 1.0, $CHCl_3$); MS (M^+) 451. Fractional crystallization from absolute ethanol afforded **4C** in 85% yield: mp 251–253 °C; $[\alpha]^{20}_D$ = −388° (*c* =).K, $CHCl_3$). From the filtrate, **4D** was obtained in 70% yield: mp 267–269 °C; $[\alpha]^{20}_D$ = +379° (*c* = 0.5, $CHCl_3$). **4C** and **4D** give the same ^{31}P NMR and ^1H NMR data as **4A** and **4B**. Elemental analysis further proved their identity and purity.

It is found that in boiling ethanol, the solubility of **4B** is 6.7 g/100 mL. Upon cooling to 25 °C, ethanol clathrated crystals are formed slowly: mp 120–121 °C dec; ^1H NMR ($CDCl_3$) δ 1.20 (t, CH_3, 3 H), 1.58 (d, CH_3, 3 H), 1.72 (s, OH, 1 H), 3.02–3.44 (m, NH, 1 H), 3.68 (q, CH_2, 2 H), 6.64–8.04 (m, Ar-H, 17 H). Anal. Calcd for $C_{28}H_{22}O_3PN{\cdot}C_2H_5OH$: C, 72.41; H, 5.68; N, 2.82. Found: C, 72.41; H, 5.54; N, 2.92. **4D** gave similar results: mp 120–121 °C dec. Anal. Calcd for $C_{28}H_{22}O_3PN{\cdot}C_2H_5OH$: C, 72.41; H, 5.68; N, 2.82. Found: C, 72.17; H, 5.61; N, 2.56. **4A** and **4C** have solubilities of 2.25 g/100 mL ethanol at 25 °C, and no clathrate formation is observed.

Reduction of 4A. Via the literature procedure,[1] **4A** resulted in a 92% yield of colorless crystalline (−)-*S*-**1**: mp 206–207 °C; $[\alpha]^{20}_D$ − 35.2° (*c* = 1.0, THF); enantiomeric purity 100%. The overall yield based upon the racemic binaphthol was 70.9%. **4B**, **4C**, and **4D** were reduced similarly, giving colorless crystalline (+)-*R*-**1**, (+)-*R*-**1**, and (−)-*S*-**1**. Their melting points, optical rotations, enantiomeric purities, and yields are summarized in the table. Phenethylamine was recovered in 80% yield with its original enantiomeric purity.

Determination of Enantiomeric Purity. The enantiomeric purity of the resolved **1** was assessed by ^{31}P NMR nonequivalence of its diastereomeric salts.[9] The (±)-binaphthyldithiophosphoric

(8) Targett, N. M.; Kilcoyne, J. P.; Green, B. *J. Org. Chem.* **1979**, *44*, 4962.

acid (dithio-BNP) was prepared in the following manner.[10] A mixture of 2,2'-dihydroxy-1,1'-binaphthyl (28.6 g, 0.10 mol) and phosphorus pentasulfide (11.0 g, 0.05 mol) was refluxed in dry xylene (200 mL). After the P_2S_5 dissolved completely, the reaction mixture was refluxed for 0.5 h and then kept for 48 h at room temperature. The crude product was collected in nearly quantitative yield. When it was recrystallized from dry toluene, colorless prisms were obtained (32.3 g, 85% yield). Anal. Calcd for $C_{20}H_{13}O_2S_2P$: C, 63.14; H, 3.45. Found: C, 63.29; H, 3.31. mp 232-234 °C; MS ($M^+ + 1$) 381; ^{31}P NMR ($CDCl_3$) 97.03 ppm. The ^{31}P NMR spectrum ($CDCl_3$) of its cinchonine salt showed resonances at 129.56 and 129.89 ppm. The *R*-dithio-BNP and *S*-dithio-BNP were prepared in the same manner using (*R*)-(+)-binaphthol ($[\alpha]_D$ +35.2°) and (*S*)-(-)-binaphthol ($[\alpha]_D$ -35.2°, respectively, as starting materials. After removal of solvent under reduced pressure, the crude acid was neutralized with an equivalent amount of cinchonine. In each case, the ^{31}P NMR spectrum ($CDCl_3$) of te salt, without any purification, showed a single signal only, indicating the 100% enantiomeric purity of each binaphthol.

(9) Mikolajezk, M.; Omelaneznk, J.; Leitloff, M.; Drabowicz, J.; Fjchart, A.; Jurczak, J. *J. Am. Chem. Soc.* **1978**, *100*, 7003.
(10) Hu, B.-F.; Sheng, Q.-F.; Li, Z.-M. *Phosphorus Sulfur* **1988**, *35*, 371.

Attempted Synthesis of Furanocyclobutenes from γ-Ketocyclobutanones

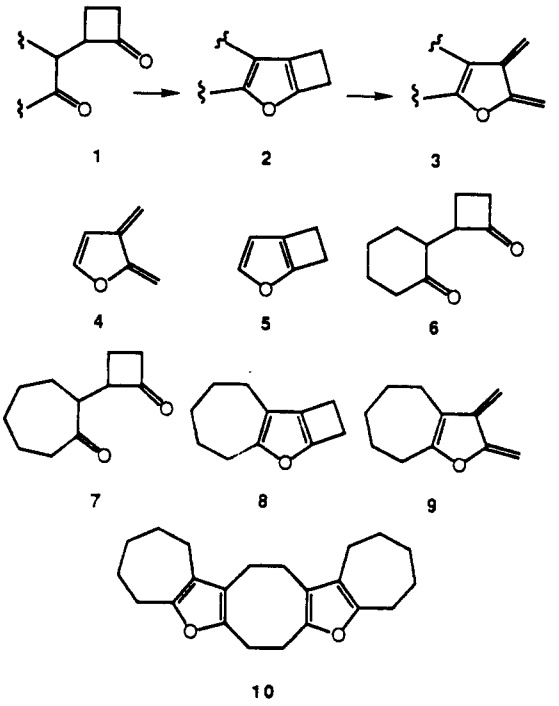
Janet A. Kaydos and Thomas A. Spencer*

Department of Chemistry, Dartmouth College, Hanover, New Hampshire 03755

Received August 14, 1989

One of the classical methods for the synthesis of furans is Paal-Knorr dehydration of enolizable 1,4-dicarbonyl compounds.[1] If such a procedure could be successfully applied to γ-ketocyclobutanones[2,3] (**1**), the products would be furanocyclobutenes (**2**). At the time this investigation was undertaken, it seemed highly probable that no successful preparation of any furanocyclobutene had been achieved.[4] There were, however, several reports of syntheses of the ring-opened isomeric 2,3-dimethylene-2,3-dihydrofurans (**3**),[5-7] and it was anticipated on the basis of the exclusive formation of **3** in all cases that if **2** were indeed formed from **1** it would spontaneously isomerize to the more stable **3**.[8] Recently, the first unequivocal synthesis of a furanocyclobutene was reported by Münzel and Schweig,[9] who photolyzed 2,3-dimethylene-2,3-dihydrofuran (**4**) in an argon matrix to afford the parent **5**.

Efforts to apply the Paal-Knorr approach to the synthesis of furanocyclobutenes were undertaken with γ-ketocyclobutanones **6** and **7**.[3] As noted above, if **7**, for example, could be converted to furanocyclobutene **8**, ring opening to **9** and formation of polymer and dimers such as **10** might be expected by analogy to results obtained by Trahanovsky.[7] In the event, treatment of **7** with *p*-toluenesulfonic acid (*p*-TsOH) in benzene at reflux for 24 h afforded mostly unreacted **7** plus a less polar, rather unstable material which intriguingly did have spectroscopic properties suggesting that it might contain some furan. The complex ^{13}C NMR spectrum of this crude product had peaks at ca. 110 ppm and at ca. 140 ppm, consistent with the presence of β and α furan carbon atoms, respectively.[10] Furthermore, bands in the IR spectrum at 1450 and 1560 cm^{-1} were reminiscent of signals previously noted in the spectra of tetrasubstituted furans.[11]



Instead of seeking initially to purify and identify this product, it was decided to run the dehydration reaction in the presence of a dienophile to trap diene **9** if indeed it were formed. A variety of dienophiles was examined, but most met with little success. The best results were obtained by using *N*-phenylmaleimide (**11**). When **7** was refluxed in toluene containing excess **11** and a small amount of *p*-TsOH, 33% of a product with mp 154.5-155.5 °C was isolated for which spectroscopic data (IR 1695, 1760 cm^{-1}; ^1H NMR 7.5 ppm, 5 H) indicated incorporation of **11**.

However, the spectral data also showed clearly that this product was not the desired adduct **12** which would be formed by reaction of **11** with **9**. In addition to the five phenyl protons there were three more ^1H NMR signals in the δ 6.0-7.9 range due to vinyl and/or aromatic protons. Surprisingly, there were also a quartet (δ 3.18, J = 8 Hz)

(1) Paal, C. *Chem. Ber.* **1884**, *17*, 2756. Knorr, L. *Chem. Ber.* **1884**, *17*, 2863.
(2) Byers, J. H.; Spencer, T. A. *Tetrahedron Lett.* **1985**, *26*, 717.
(3) Kaydos, J. A.; Byers, J. H.; Spencer, T. A. *J. Org. Chem.* **1989**, *54*, 4698.
(4) Ruhlmann, K.; Kokkali; A.; Berker, H.; Seefluth, J.; Frieden, U. *J. Prakt. Chem.* **1969**, *311*, 844 claim the synthesis of 2-amino-3-cyanofuranocyclobutene. However, since the only data reported for this substance are melting point and elemental analysis, and since there is good reason to regard furanocyclobutenes as highly unstable (refs 5-9), it seems reasonable to question the structural assignment.
(5) Winberg, H. E.; Fawcett, F. S.; Mochel, W. E.; Theobald, C. W. *J. Am. Chem. Soc.* **1960**, *82*, 1428.
(6) Jullien, J.; Pechine, J. M.; Perez, F.; Piade, J. J. *Tetrahedron Lett.* **1979**, 3079.
(7) Trahanovsky, W. S.; Cassady, T.; Woods, T. L. *J. Am. Chem. Soc.* **1981**, *103*, 6691.
(8) MNDO calculations (ref 8), as well as the failure to detect any **2** experimentally (refs 4-6), indicate that **3** is significantly more stable than **2**.
(9) Münzel, N.; Schweig, A. *Angew. Chem., Int. Ed. Engl.* **1987**, *26*, 471.
(10) Silverstein, R. M.; Bassler, G. C.; Morrill, T. C. *Spectrometric Identification of Organic Compounds*, 4th ed.; John Wiley & Sons: New York, 1981; p 297, report ^{13}C NMR δ values of 142.8 and 109.7 ppm for the α and β carbons of furan.
(11) Unpublished data concerning compounds described in: Spencer, T. A.; Britton, R. W.; Watt, D. S. *J. Am. Chem. Soc.* **1967**, *89*, 5727.

0022-3263/91/1956-0425$02.50/0