NCS–Br_2 Reaction. CH_2Cl_2. In a typical experiment a mixture of 0.24 mmol of NCS, 0.045 mmol of Br_2, and 7.78 mmol of CH_2Cl_2 was irradiated with visible light at 15 °C. After 15 min the solution was still colored. By NMR analysis 19% of the NCS had reacted and $CHCl_2Br$ could be detected. On further irradiation the solution became colorless, and when all the NCS was consumed, analysis indicated 0.16 mmol of $CHCl_3$ and 0.081 mmol of $CHCl_2Br$ (90% based on Br_2). In a similar experiment with equal quantities of Br_2 and NCS, $CHCl_2Br$ was the sole halogenated product detected.

Neopentane. Similar photolysis of 0.30 mmol of NCS, 0.49 mmoles of Br_2, 1.88 mmol of neopentane, and 12.3 mmol of CH_2Cl_2 showed complete consumption of NCS in 10 min, with neopentyl bromide the only product detected by NMR (essentially quantittive based on NCS). Several other experiments, using up to 10 mmol of NCS and equivalent amounts of Br_2 gave similar results.

***n*-Butane.** Similar photolysis of 0.32 mmol of NCS, 0.27 mmol of Br_2, and 1.47 mmol of *n*-butane in 7.59 mmol of CH_2Cl_2 was complete in 15 min. NMR analysis showed both *sec*- and *n*-butyl bromides, and GLC analysis showed peaks agreeing with authentic samples: areas (*sec*-butyl:*n*-butyl) 1.89:1.

Cyclopropane. A similar reaction with cyclopropane gave a very rapid reaction. GC analysis showed chiefly 1,3-dibromopropane with no evidence of cyclopropyl bromide.

Acknowledgment. Partial support of this work by a grant from the National Science Foundation is gratefully acknowledged.

Registry No. NCS, 128-09-6; DCE, 75-35-4; Br_2, 7726-95-6; CH_2Cl_2, 75-09-2; $CHCl_2Br$, 75-27-4; $CHCl_3$, 67-66-3; HCl, 7647-01-0; β-chloropropionyl isocyanate, 54898-89-4; β-chloropropionamide, 5875-24-1; neopentane, 463-82-1; neopentylbromide, 630-17-1; *n*-butane, 106-97-8; *sec*-butyl bromide, 78-76-2; *n*-butyl bromide, 109-65-9; 1,3-dibromopropane, 109-64-8; cyclopropane, 75-19-4.

A Strained Cyclopropanobicyclo[3.2.1]octanone

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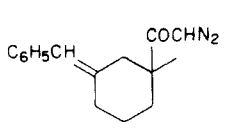
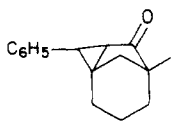
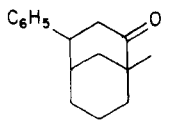
A short 1976 communication[1] descirbed the preparation of tricyclic ketone **2** via the decomposition of diazo ketone **1** under the influence of light, heat, and cupric oxide and the conversion of the product into bicyclic ketone **3** on reduction with lithium in ammonia. These observations are so unexpected, bordering on disbelief,[2] as to invite repetition of the experiments. The first reaction involved the formation of an extraordinarily strained ring system in a diazo ketone decomposition in the face of alternate, low-energy decomposition routes.[3] The second reaction, considered unusual even by the authors (as illustrated by the communication's title, "Unusual Ring Opening of Conjugated Phenylcyclopropyl Ketone; A New Route to Bicyclo[3:3:1]nonane Intermediate", and by their early comments[1]), constituted a violation of the well-known lithium–ammonia reduction mode of cyclopropyl ketones, i.e., cleavage of the cyclopropane bond most closely paralleling the neighboring keto π orbital.[4] The present paper represents an experimental reevaluation of the earlier work.



1

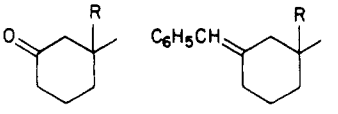


2



3

The preparation of the diazoketone had been initiated by a Wittig reaction with benzyltriphenylphosphorane on a keto acid (**4b**)[5] of unspecified origin.[1] To facilitate the reaction scheme, the Wittig reaction now was carried out on keto nitrile **4a**,[5] prepared by the hydrocyanation of 3-methyl-2-cyclohexenone.[6] Alkaline hydrolysis of the resultant olefinic cyanide isomers (**5a**) led to a difficultly separable, ca. 1:1 stereoisomer mixture of acids **5b**. In order to make the first comparison with the literature data, the acids were transformed into the esters **5c** with diazomethane. The olefinic ester mixture had been reported to exhibit the following spectral data:[1] UV (EtOH) λ_{max} 250 nm (ε 20 040);[7] IR ($CHCl_3$) 1725, 1600 cm^{-1}; ^1H NMR (CCl_4) δ 1.1, 1.2 (3 H, 2 s from C-3 methyl protons), 3.42, 3.62 (3 H, 2 s from CO_2Me protons), 6.2 (1 H, s) 7.13 (5 H, s). It now revealed the ensuing data: UV (EtOH) $\lambda_{shoulder}$ 227–250 nm; IR ($CHCl_3$) 1711 (s, C=O), 1590 (w, C=C) cm^{-1}; ^1H NMR δ (CCl_4) 1.03, 1.18 (s, 3 total, Me of each isomer), 3.44, 3.53 (s, 3 total, OMe of each isomer), 5.29, 5.38 (s, 1 total, olefinic H of each isomer), 7.00 (br.s, 5, Ar H). The two sets of data were divergent, albeit not dramatically.



4a, R=CN
b, R=CO_2H

5a, R=CN
b, R=CO_2H
c, R=CO_2Me

Treatment of the acids **5b** with oxalyl chloride and the resultant acid chlorides with diazomethane yielded diazo ketones **1** [IR ($CHCl_3$) 2113 (s, CHN_2), 1622 (s, C=O), 1600 (w, C=C) cm^{-1} (lit.[1] IR ($CHCl_3$) 2110, 1600 cm^{-1})]. Decomposition of the latter under the literature conditions led in 41% yield to a five-membered-ring $C_{16}H_{18}O$ ketone [IR ($CHCl_3$) 1700 (s, C=O), 1610 (w, C=C) cm^{-1} (lit.[1] IR ($CHCl_3$) 1705, 1600 cm^{-1}); MS, *m/e* (relative intensity) 226 (M^+, 9), 225 (41), 135 (34), 107 (53), 91 (73), 43 (base) (lit.[1] MS, *m/e* 226], whose ^1H NMR spectrum [δ (CCl_4) 0.90 (s, 3, Me), 2.69, 2.84, 2.92, 3.07 (four-line AB, 2, J = 14 Hz, *c*-Pr H), 6.9–7.2 (m, 5, Ar H)] was compatible with structure **2** but in disagreement with the published ^1H NMR spectral results [δ (CCl_4) 0.78 (1 H, s, cyclopropane H), 0.93 (3 H, s), 3.33 (1 H, m, benzyl H), 7.16 (5 H, s)][1]. The ^{13}C NMR spectrum (carbon shifts being listed on

(1) Sarkar, A.; Chatterjee, S.; Dutta, P. C. *Tetrahedron Lett.* **1976**, 4633.
(2) The credibility of the work was lowered on discovery of the untrustworthiness of a later publication (Chatterjee, S. *Tetrahedron Lett.* **1979**, 3249) of one of the coauthors as an independent investigator (see: Cornforth, Sir John *Ibid.* **1980**, 709. Cornforth, Sir John; Pengelly, T. *Ibid.* **1982**, 2213) and the dubiety of some of the results of another paper (Chatterjee, S. *J. Chem. Soc., Chem. Commun.* **1979**, 620) by the same contributor (see: Paquette, L. A.; Han, Y.-K. *J. Org. Chem.* **1979**, *44*, 4014; *J. Am. Chem. Soc.* **1981**, *103*, 1835).
(3) Wenkert, E.; Davis, L. L.; Mylari, B. L.; Solomon, M. F.; da Silva, R. R.; Shulman, S.; Warnet, R. J.; Ceccherelli, P.; Curini, M.; Pellicciari, R. *J. Org. Chem.* **1982**, *47*, 3242.
(4) Dauben, W. G.; Deviny, E. J. *J. Org. Chem.* **1966**, *31*, 3794.
(5) Whitmore, W. F.; Roberts, C. W. *J. Org. Chem.* **1948**, *13*, 31.
(6) Cf.: Wenkert, E.; Strike, D. P. *J. Am. Chem. Soc.* **1964**, *86*, 2044.
(7) Benzylidenecyclohexane has been reported to exhibit a UV absorption maximum in ethanol solution at 247 nm (log ε 4.04) (Christol, H.; Laurent, A.; Mousseron, M. *Bull. Soc. Chim. Fr.* **1961**, 2313).

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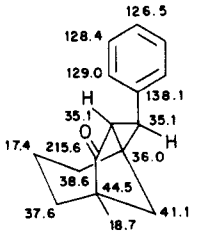
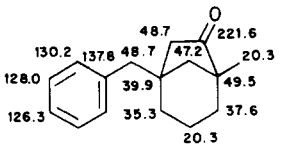
formula 6) showed the material to be a single compound[8] and to possess structure 2 (=6).[9] Decomposition of the



6

diazo ketones 1 under the influence of the modern cyclopropanation catalyst, dirhodium tetraacetate, produced the same tricyclic ketone in 31% yield.

Reduction of ketone 6 with lithium in ammonia for a short time afforded the 6-bicyclo[3.2.1]octanone 7 in 90% yield. The structure of the compound could be identified from the infrared [($CHCl_3$) 1730 (s, C=O), 1601 (w, C=C) cm^1 (lit.[10] IR ($CHCl_3$) 1725, 1600 cm^{-1})][11] and ^1H NMR [δ (CCl_4) 0.95 (s, 3, Me), 2.00 (s, 2, Ar CH_2), 2.68 (s, 2, $COCH_2$), 6.9–7.2 (m, 5, Ar H) (lit.[10] ^1H NMR δ (CCl_4) 0.95 (3 H, s), 2.0 (2 H, s, $COCH_2$), 2.66 (2 H, s, Ar CH_2), 7.2 (5 H, s)] spectra and corroborated by the ^{13}C NMR spectrum (carbon shifts cited on formula 7).[12] This result stands in sharp contrast to the claim[1] of the reduction product being represented by structure 3 and being nonidentical with ketone 7 but is in accord with expectations for the lithium–ammonia reduction of α,β-cyclopropano ketones.[4]



7

The ease of formation of structurally complex tricyclic ketones of type 2 by the decomposition of easily accessible γ,δ-unsaturated diazo ketones bodes well for their future use in organic synthesis.

Experimental Section

Ultraviolet spectra of ethanol solutions and infrared spectra of chloroform solutions were obtained on Perkin-Elmer 551S and 1320 spectrophotometers, respectively. ^1H NMR spectra of $CDCl_3$ solutions were recorded on a Varian EM-390 spectrometer and ^{13}C NMR spectra of $CDCl_3$ solutions on a Bruker WP 80 sy spectrometer operating at 20.15 MHz in the Fourier transform mode. The carbon shifts are in parts per million downfield from Me_4Si; $\delta(Me_4Si) = \delta(CDCl_3) + 76.9$ ppm. Elemental analyses were acquired on a Carlo Erba Model 1106 elemental analyzer. Column chromatography was carried out on 0.063–0.200 mesh Merck silica gel. All extracts were dried over Na_2SO_4.

3-Cyano-3-methylcyclohexanone (4a). A solution of 4.50 g (41 mmol) of 3-methyl-2-cyclohexenone in 100 mL of dimethylformamide was treated with a solution of 6.30 g (97 mmol) of potassium cyanide and 4.50 g (85 mmol) of ammonium chloride in 130 mL of water and the mixture stirred at 100 °C for 5 h. It then was concentrated to a syrup under vacuum at 60 °C, diluted with 100 mL of water, and extracted exhaustively with ether. The extract as dried ($MgSO_4$) and evaporated. The residue, liquid cyano ketone **4a** [IR 2251 (m, C≡N), 1722 (s, C=O) cm^{-1}; ^1H NMR δ 1.48 (s, 3, Me), 2.22, 2.39, 2.62, 2.79 (four-line AB, 2, J = 13 Hz, C-2 H)],[5] was used in the next reaction without further purification.

Anal. Calcd for $C_8H_{11}ON$: C, 70.04; H, 8.08; N, 10.21. Found: C, 70.12; H, 8.01; N, 10.32.

3-Benzylidene-1-cyano-1-methylcyclohexane (5a). Dry tetrahydrofuran (80 mL) was added to a mixture of 1.78 g (33 mmol) of solid sodium methoxide and 8.52 g (22 mmol) of benzyltriphenylphosphonium chloride and the mixture stirred under nitrogen for 20 min. A solution of 3.00 g (22 mmol) of ketone **4a** in 20 mL of dry tetrahydrofuran was added to the orange-red suspension and the mixture refluxed under nitrogen for 6 h. It then was poured into 50 mL of water and extracted with chloroform. The extract was dried and evaporated. Chromatography of the residue and elution with 25:1 hexane–ethyl acetate gave 2.40 g (52%) of liquid nitrile **5a**: ^1H NMR δ[13] [syn isomer] 1.34 (s, 3, Me), 1.87, 2.02, 2.88, 3.03 (four-line AB, 2, J = 14 Hz, C-2 H), 6.48 (br s, 1, olefinic H), 7.1–7.3 (m, 5, Ar H), [anti isomer] 1.40 (s, 3, Me), 2.10, 2.24, 2.50, 2.64 (four-line AB, 2, J = 14 Hz, C-2 H), 6.36 (br s, 1, olefinic H), 7.1–7.3 (m, 5, Ar H).

Anal. Calcd for $C_{15}H_{17}N$: C, 85.26; H, 8.11; N, 6.63. Found: C, 85.12; H, 8.21; N, 6.68.

3-Benzylidene-1-methylcyclohexanecarboxylic Acid (5b). A suspension of 10.60 g (200 mmol) of powdered potassium hydroxide and 1.20 g (6 mmol) of nitrile **5a** in 130 mL of diethylene glycol was heated at 200 °C under nitrogen for 12 h. The cooled mixture was poured into 100 mL of water and extracted with chloroform. The aqueous solution was brought to pH 2 with 4% sulfuric acid solution and extracted with chloroform. The extract was washed with water, dried, and evaporated. The residue, liquid olefinic acid **5b** [IR 3100–3500 (m, OH), 1702 (s, C=O), 1600 (w, C=C) cm^{-1}; ^1H NMR δ 1.20, 1.32 (s, 3 total, Me of each isomer), 5.42, 5.52 (br s, 1 total, olefinic H of each isomer), 7.1–7.3 (m, 5, Ar H); MS, m/e (relative intensity) 230 (M^+, 62), 185 (base), 91 (83); ^{13}C NMR δ [syn isomer] 19.9 (C-5), 26.6 (Me), 27.6 (C-6), 32.6 (C-4), 41.3 (C-1), 44.5 (C-2), 184.5 (C=O), [anti isomer] 22.7 (C-5), 23.8 (Me), 30.6 (C-6), 36.9 (C-4), 43.4 (C-1), 44.4 (C-2), 183.9 (C=O)], was used in the next reaction without further purification.

Anal. Calcd for $C_{15}H_{18}O_2$: C, 78.23; H, 7.88. Found: C, 78.31; H, 7.73.

5-Methyl-2-phenyl-4-tricyclo[3.3.1.0^{1,3}]nonanone (2, 6). A solution of 500 mg (2 mmol) of acid **5b** in 5 mL of freshly distilled oxalyl chloride was stirred at room temperature under nitrogen for 4 h and the excess reagent then removed by distillation under vacuum. A solution of the residue in 20 mL of dry ether was added to a stirring 0.02 M ethereal diazomethane solution (150 mL) containing 0.3 mL (2 mmol) of triethylamine under nitrogen at 0 °C and the stirring continued at room temperature for 3 h. The mixture was filtered and the filtrate evaporated under vacuum. The residue, diazo ketone 1 [^1H NMR δ (both isomers) 1.06, 1.19 (s, 3 total, Me), 5.10, 5.38 (s, 1 total, CHN_2), 5.34, 5.47 (s, 1 total, olefinic H), 7.0–7.3 (m, 5 total, Ar H)], was used in the next reaction without further purification.

Copper oxide,[14] 100 mg, was added to a stirring solution of 300 mg (1.2 mmol) of diazo ketone 1 in 45 mL of dry tetrahydrofuran and 105 mL of dry cyclohexane under nitrogen and the suspension refluxed and irradiated with a 300-W tungsten lamp for 22 h. Throughout this period three more 100-mg samples of copper oxide were added. Then the mixture was filtered through a fritted glass funnel and the filtrate evaporated. Chromatography of the residue and elution with benzene yielded 110 mg (41%) of liquid tricyclic ketone 2 (6): ^1H NMR δ 0.90 (s, 3, Me), 2.68, 2.84, 2.88, 3.04 (four-line AB, 2, J = 14 Hz, cyclopropane H), 7.0–7.3 (m, 5, Ar H).

(8) The formation of only one stereoisomeric cyclopropane derivative may have been due to (a) its isomer not having survived the reaction conditions or (b) the isolated cyclopropane being the more stable isomer of the two highly strained tricycles and rotation of the bond between the benzyl and β-phenylethyl carbons having taken place at some time during the multistage cyclopropanation process.

(9) The stereochemistry of the phenyl substitution site is considered only as tentative.

(10) Sarkar, A.; Mukherjee, D. *Indian J. Chem., Sect. B* **1976**, *14B*, 798.

(11) The most frequently quoted infrared carbonyl stretching frequency for 6-bicyclo[3.2.1]octanones is 1735 cm^{-1} (e.g.: Bell, R. A.; Ireland, R. E.; Partyka, R. A. *J. Org. Chem.* **1966**, *31*, 2530. Mukherjee, D.; Mukhopadhyay, S. K.; Mahalanabis, K. K.; Das Gupta, A.; Dutta, P. C. *J. Chem. Soc., Perkin Trans. 1* **1973**, 2083).

(12) Cf.: Stothers, J. B.; Swenson, J. R.; Tan, C. T. *Can. J. Chem.* **1975**, *53*, 581.

(13) Various chromatographic fractions containing different proportions of the two stereoisomers permitted their differentiation.

(14) Ghatak, U. R.; Chakraborty, P. C.; Ranu, B. C.; Sanyal, B. *J. Chem. Soc., Chem. Commun.* **1973**, 548 and reeferences therein.

Anal. Calcd for $C_{16}H_{18}O$: C, 84.91; H, 8.02. Found: C, 84.76; H, 8.13.

6-Bicyclo[3.2.1]octanone 7. Lithium (20 mg, 3 mmol) was dissolved in 40 mL of liquid ammonia at −40 °C under nitrogen, and a solution of 100 mg (0.4 mmol) of ketone **2** (**6**) in 5 mL of dry tetrahydrofuran was added over a 5-min period to the stirring mixture. The stirring was continued for 10 min. Enough ammonium chloride was added to discharge the color of the mixture and the ammonia was allowed to evaporate. Sulfuric acid solution, 25 mL of 2%, was added and the mixture extracted with chloroform. The extract was washed with water, dried, and evaporated. Chromatography of the residue and elution with 50:1 hexane–ethyl acetate gave 90 mg (90%) of liquid ketone **7**: ^{1}H NMR δ 0.99 (s, 3, Me), 2.10 (s, 2, benzyl H), 2.72 (s, 2, $COCH_2$), 7.0–7.4 (m, 5, Ar H).

Anal. Calcd for $C_{16}H_{20}O$: C, 84.16; H, 8.83. Found: C, 84.06; H, 8.98.

Acknowledgment. P.C., M.C., and M.C.M. are indebted to the Centro Nazionale delle Ricerche (Rome) and the Ministero della Pubblica Istruzione for financial support.

Registry No. *syn*-**1**, 99885-24-2; *anti*-**1**, 99885-25-3; **2**, 62701-58-0; **3**, 62733-87-3; **4a**, 33235-14-2; *syn*-**5a**, 99885-18-4; *anti*-**5a**, 99885-19-5; *syn*-**5b**, 99885-20-8; *anti*-**5b**, 99885-21-9; *syn*-**5b** (acid chloride), 99885-22-0; *anti*-**5b** (acid chloride), 99885-23-1; *syn*-**5c**, 99885-27-5; *anti*-**5c**, 99885-28-6; **7**, 99885-26-4; 3-methyl-2-cyclohexenone, 1193-18-6; benzyltriphenylphosphonium chloride, 1100-88-5.

Solid–Liquid Phase-Transfer Catalysis without Solvent: Mild and Efficient Conditions for Saponifications and Preparations of Hindered Esters

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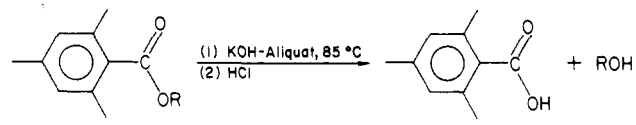
Recently, we reported that, when used in the absence of organic solvent, the "solid KOH–Aliquat 336 (2%) system" was an efficient basic agent which allowed generating in situ (for subsequent alkylations) oxyanions from acids[1] and aliphatic[2] or aromatic alcohols[3,4] and which also promoted β-elimination from secondary halides.[5]

In this work, we attempted to test the efficacy of this basic system for the difficult problem of hindered ester hydrolysis (saponifications). To this purpose, the hydrolysis of mesitoic esters constitutes a classical test to evaluate the ability of a basic system to act as a strong nucleophile toward an ester carbonyl group. This study is of prime interest for a great need still exists for efficient and mild methods, since current procedures suffer from many disadvantages connected to poor yields or cost and toxicity of solvents and catalysts (crown ethers, cryptates, ...).

In the course of the present study, we have extended some previous experiments on carboxylate alkylations[1] to the improvement of hindered ester synthesis also in need of efficient and mild methods[6,7] since a number of useful and reliable methods are not suitable in this case.

Table I. Saponification of Mesitoic Esters 1a and 1d



(10 mmol)

1a, R = CH_3
d, R = n-C_8H_{17}

ester	mol equiv of KOH	time, h	isolated yield, %
1a	2	5	70
	5	2	80
	5	5	93
1d	5[a]	24	80
	5	2	53
	5	5	87[b]

[a] 60 °C. [b] An isolated yield of 83% in n-octanol was obtained.

Results and Discussion

Saponification of Mesitoic Esters. It was shown using oxygen-18 labeled experiments that, even in the absence of organic solvent, the mechanism consists of a nucleophilic attack by hydroxide ion on the carbonyl carbon of mesitoic esters.[11]

Thus, in our hands, methyl and octyl mesitoate (**1a** and **1d**) saponifications have been performed without organic solvent in the presence of powdered KOH (as a commercial base containing about 15% water) and 2% Aliquat 336[8] which mainly consists of methyltrioctylammonium chloride. These are assumed to be solid–liquid phase-transfer catalysis (PTC) conditions where neat esters constitute the organic liquid phase. The main results are listed in Table I.

Our attempts at saponification of hindered esters **1a** and **1d** are very fruitful, and very good yields of acids are obtained (93% and 87%, respectively). The best results are reached when the saponifications are performed at 85 °C for 5 h using 5 mol equiv of powdered KOH + 2% Aliquat 336 (third and sixth entries).

In order to appreciate the efficiency of our basic system, we have collected in Table II the best results described until now. It is clear from this table that the "solid KOH–Aliquat" system is very attractive: (1) From a reactivity point of view, it is one of the most efficient for obtaining good yields under rather mild conditions. These conditions constitute a large improvement when compared to the literature methods which need stoichiometric amounts of crown ethers or cryptands in toluene[9,10] or the prior and rather difficult preparations of reagents such as "anhydrous KOH"[11] or PEG grafted to a cross-linked polystyrene.[12] However, it appears that the KOH/Me_2SO

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