ated phosphine. A possible sequence of events to account for the product is shown above. The transesterification may occur before or after the ring cleavage.

Experimental Section

General. Reactions of phosphines were conducted under nitrogen. Mp values are corrected. Proton nmr spectra were taken with JEOL MH-100 or Varian T-60 spectrometers. Proton-decoupled ^{31}P nmr spectra were taken on a Bruker HFX-10 system at 36.43 MHz; shifts are relative to external 85% H_3PO_4. The proton-decoupled ^{13}C nmr spectrum was obtained by the Fourier transform technique on the Bruker spectrometer at 22.62 MHz utilizing C_6F_6 as external heteronuclear lock in a 3-mm coaxial capillary. Analyses were performed by MHW Laboratories, Garden City, Mich.

Synthesis of Methyl(4-carboxybutyl)phosphine Oxide (3) from Methyl 1-Methylphospholane-2-carboxylate (1). To 30 ml of deoxygenated 91% formic acid was added 0.40 g (0.0025 mol) of a 60:40 cis:trans mixture of 1.[2] Dry hydrogen chloride generated from sodium chloride and concentrated sulfuric acid was bubbled for 10 min through the resulting solution, which was then refluxed for 24 hr under nitrogen. The reflux condenser was equipped with a take-off valve, and a total of 10 ml of distillate containing the methyl formate produced was drawn off during the reaction period. After the reflux period was complete, the formic acid was distilled off at water-aspirator pressure. Water was added and the distillation repeated to remove remaining traces of formic acid. A light brown oil remained which solidified upon drying overnight at high vacuum. This solid was recrystallized from chloroform–petroleum ether and yielded 0.29 g (71%) of white crystalline **3**, mp 87–88°.

The ^1H nmr spectrum (external TMS) gave the following signals: in H_2O, δ 2.15 (d of d, *P*-CH_3, $^2J_{PH}$ = 14 Hz, $^3J_{HH}$ = 2 Hz), 1.9–2.7 and 2.8–3.2 (multiplets, CH_2), δ 9.98 (half of doublet of sextets with other half under H_2O absorption, *P*-H, $^3J_{HH}$ = 2 Hz); in D_2O, δ 2.15 (d, *P*-CH_3, $^2J_{PH}$ = 14 Hz), 9.98 was absent, rest unchanged; in $CDCl_3$ δ 1.9–2.7 and 2.7–3.3 (two broad peaks, indistinct *P*-CH_3 and CH_2), δ 7.62 (d of broad peaks, *P*-H, $^1J_{PH}$ = 476 Hz), 10.31 (broad s, COOH). The ^{31}P nmr had signals at δ −38.2 in H_2O, −37.8 in D_2O (t, $^1J_{PD}$ = 73 Hz), and −31.8 in $CHCl_3$. The ^{13}C nmr (H_2O, *p*-dioxane as internal reference, δ^{TMS} = 67.8 ppm) is described in the discussion. The infrared spectrum (KBr disk) contained absorptions at 2550, 2900, and 1925 for hydrogen bonded OH stretch, ν_{PH} 2375, $\nu_{C=O}$ 1700, $\nu_{P=O}$ 1110 cm^{-1}.

Anal. Calcd for $C_6H_{13}O_3P$: C, 43.91; H, 7.99; P, 18.87. Found: C, 43.60; H, 7.93; P, 18.56.

Cleavage of 1-Methyl-3-phospholanone (5). By the same procedure as above, 0.9 g (0.0073 mol) of **5**[9] was treated with formic acid–HCl. A light green oil was obtained after removal of all the formic acid. Addition of chloroform dissolved most of the oil leaving a small amount of green residue. The chloroform was removed by rotary evaporation, which yielded 0.52 g (51%) of **6** as a thick, almost colorless oil. All attempts to crystallize the oil proved unsuccessful.

The ^1H nmr spectrum (H_2O, external TMS) gave the following signals: in H_2O, δ 2.03 (d of d, *P*-CH_3, $^2J_{PH}$ = 14 Hz, $^3J_{HH}$ = 4 Hz), 2.4–3.0 (m, CH_2), 2.61 (s, CH_3CO), 10.02 (broad signal with indistinct additional splitting, half of *P*-H doublet); in D_2O, δ 2.02 (d, *P*-CH_3, $^2J_{PH}$ = 14 Hz), 10.02 was absent, rest unchanged; in $CDCl_3$, δ 2.16 (broad d, *P*-CH_3, $^2J_{PH}$ = 13–14 Hz), 2.4–3.0 (m, CH_2), 2.75 (s, CH_3CO), 7.72 (broad d, *P*-H, $^1J_{PH}$ = 474 Hz). The ^{31}P nmr signal was at δ −38.2 in H_2O, −37.0 in D_2O (t, $^1J_{PD}$ = 76 Hz) and −27.5 in $CDCl_3$. The ir spectrum (neat) had $\nu_{C=O}$ 1720 and $\nu_{P=O}$ 1155 cm^{-1}.

Spectra of 3,4-Dimethyl-3-phospholene 1-Oxide (4). This compound was prepared as previously reported.[5] The ^1H nmr spectrum (external TMS) had the following signals: in H_2O, δ 2.22 (s, *C*-CH_3), 2.78–3.50 (m, CH_2), 10.35 (broad, half of *P*-H doublet, removed with D_2O); in $CDCl_3$ the PH signal occurred at δ 7.97 (J_{PH} = 490 Hz). The ^{31}P nmr signal was at δ −44.4 in H_2O, −44.1 in D_2O (t, $^1J_{PD}$ = 76 Hz), and −39.8 in $CDCl_3$.

Registry No.—*cis*-**1**, 52500-00-2; *trans*-**1**, 52500-01-3; **3**, 52571-12-7; **4**, 52500-02-4; **5**, 49849-35-6; **6**, 52571-13-8.

References and Notes

(1) Supported by Public Health Service Research Grant CA-05507 from the National Cancer Institute. The National Science Foundation provided funds toward the purchase of the Bruker spectrometer (Grant No. GP-10301).
(2) S. G. Borleske and L. D. Quin, *Phosphorus*, in press.
(3) C. E. Rehberg, "Organic Syntheses," Collect. Vol. III, Wiley, New York, N.Y., 1955, p 33.
(4) L. A. Hamilton and P. S. Landis, in "Organic Phosphorus Compounds," Vol. 4, G. M. Kosolapoff and L. Maier, Ed., Wiley-Interscience, New York, N.Y., 1972, p 490.
(5) D. K. Myers and L. D. Quin, *J. Org. Chem.*, **36**, 1290 (1972).
(6) See, for example, G. A. Gray and S. E. Cremer, *J. Org. Chem.*, **37**, 3458, 3470 (1972); J. J. Breen, S. I. Featherman, L. D. Quin, and R. C. Stocks, *Chem. Commun.*, 657 (1972).
(7) R. Hagen and J. D. Roberts, *J. Amer. Chem. Soc.*, **91**, 4504 (1969).
(8) L. D. Quin, M. D. Gordon, and S. O. Lee, *Org. Magn. Resonance*, in press.
(9) L. D. Quin and R. C. Stocks, *J. Org. Chem.*, **39**, 686 (1974).

New Facile Method for Conversion of Oximes to Nitriles. Preparation and Acid-Catalyzed Transformation of Aldehyde Oxime Ortho Esters

Milorad M. Rogić,* Jan F. Van Peppen, Karl P. Klein, and Timothy R. Demmin

Chemical Research Center, Allied Chemical Corporation, Morristown, New Jersey 07960

Received May 12, 1974

We wish to report a new and facile conversion of aldoximes to the corresponding nitriles by an acid-catalyzed reaction of aldoximes and ortho esters (eq 1).

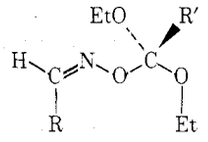
$$RCH{=}NOH + R'C(OEt)_3 \xrightarrow{H^-} RCN + R'COOEt + 2EtOH \quad (1)$$

Heating a mixture of equivalent amounts of an aldoxime and an ortho ester in the presence of a catalytic amount of an acid resulted in formation of the corresponding nitrile, ester, and alcohol. Simple distillation of the ester and the alcohol thus produced (eq 1), followed by vacuum distillation of the residue, afforded the nitrile usually in high yield. The general nature of the reaction is indicated by the results summarized in Table I. The primary product in this transformation is the oxime dialkyl ortho ester[1] which can be easily isolated in high yield by distilling off 1 equiv of the alcohol from an equimolar mixture of the oxime and the ortho ester *in the absence of acid catalysts* (eq 2).

$$RCH{=}NOH + R'C(OEt)_3 \rightleftharpoons RCH{=}NOCR'(OEt)_2 + EtOH \quad (2)$$

For example, distillation of 1 equiv of ethanol from a reaction mixture of equimolar amounts of *n*-butyraldehyde oxime (a mixture of *Z* and *E* isomers in the approximate ratio of 3:2) and triethyl orthoacetate, followed by vacuum distillation, gave a 95% yield of *n*-butyraldehyde oxime diethyl orthoacetate. Similarly, *Z*-benzaldehyde oxime and triethyl orthoformate gave an 86% yield of benzaldehyde oxime diethyl orthoformate. Analysis of these reactions *via* nmr spectroscopy indicated that no oxime isomerization had occurred under the reaction conditions.[3] The formation of oxime dialkyl ortho esters is evidently also a general reaction as indicated in Table II.

The oxime dialkyl ortho esters undergo an acid-catalyzed Beckmann fragmentation reaction providing the corresponding nitrile, ester, and alcohol[5] (eq 3). This reaction



$$\xrightarrow{H^-} RCN + R'COOEt + EtOH \quad (3)$$

Table I
Conversion of Aldehyde Oximes with Ortho Esters to Nitriles

Oxime	Registry no.	mmol	Reagent	mmol	Solvent	Nitrile	Registry no.	Yield,[a] %
$CH_3(CH_2)_2CH{=}NOH$	5780-44-6 (*Z*) 5775-75-7 (*E*)	100	$HC(OEt)_3$[b,f]	120		$CH_3(CH_2)_2CN$	109-74-0	93
$CH_3(CH_2)_2CH{=}NOH$		100	$CH_3C(OEt)_3$[b,g]	120		$CH_3(CH_2)_2CN$		94
$CH_3(CH_2)_2CH{=}NOH$		100	$CH_3C(OEt)_3$[c]	100	SO_2	$CH_3(CH_2)_2CN$		96
$CH_3(CH_2)_5CH{=}NOH$	629-31-2	100	$HC(OEt)_3$[b]	120		$CH_3(CH_2)_5CN$	629-08-3	95
$EtO_2C(CH_2)_3CH{=}NOH$	42586-31-2	50	$HC(OEt)_3$[d]	60	Et_2O	$EtO_2C(CH_2)_3CN$	10444-38-9	92
$EtO_2C(CH_2)_4CH{=}NOH$	42586-31-3	50	$HC(OEt)_3$[d]	60	$CHCl_3$	$EtO_2C(CH_2)_4CN$	4450-39-9	96
$C_6H_5CH{=}NOH$	932-90-1	100	$HC(OEt)_3$[c]	100	SO_2	C_6H_5CN	100-47-0	90
$C_6H_5CH{=}NOH$		100	$CH_3C(OEt)_3$[c]	100	SO_2	C_6H_5CN		60
p-$CH_3OC_6H_4CH{=}NOH$	3235-04-9	50	$HC(OEt)_3$[c,e]	50	SO_2	p-$CH_3OC_6H_4CN$	874-90-8	90
p-$CH_3OC_6H_4CH{=}NOH$		50	$CH_3C(OEt)_3$[c,e]	50	SO_2	p-$CH_3OC_6H_4CN$		90
$C_6H_5CH_2CH_2CH{=}NOH$	1197-50-8	50	$HC(OEt)_3$[b]	60		$C_6H_5CH_2CH_2CN$	645-59-0	93

[a] Yields are of the isolated products. [b] Reaction carried out in the presence of 2 mol % methanesulfonic acid by heating the reaction mixture in the absence of solvents. [c] Reaction carried out using the corresponding oxime diethyl ortho ester in the absence of added catalyst. [d] Reaction carried out in the presence of 2 mol % of hydrochloric acid under reflux in the indicated solvent. [e] After 78 hr at 70°. [f] Registry no. 122-51-0. [g] Registry no. 78-39-7.

Table II
Preparation of Oxime Diethyl Ortho Esters[a]

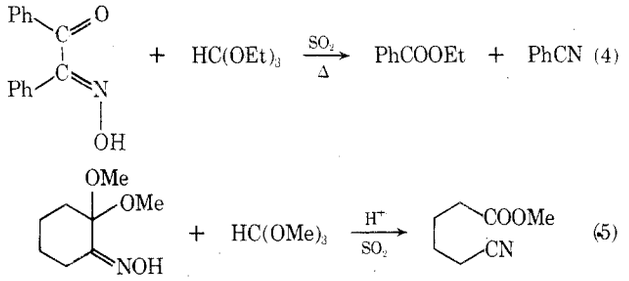
Oxime	Reagent	Product[b]	Registry no.	Bp, °C (mm)	Yield,[c] %
$CH_3(CH_2)_2CH{=}NOH$	$HC(OEt)_3$	$CH_3(CH_2)_2CH{=}NOCH(OEt)_2$	52540-27-9	67-69 (1-2)	95
$CH_3(CH_2)_2CH{=}NOH$	$CH_3C(OEt)_3$	$CH_3(CH_2)_2CH{=}NOCCH_3(OEt)_2$	52540-28-0	47-49 (0.6)	90
$CH_3(CH_2)_2CH{=}NOH$	$PhC(OEt)_3$[f]	$CH_3(CH_2)_2CH{=}NOCPh(OEt)_2$	52540-29-1	115-119 (1.1)	82
$C_6H_5CH{=}NOH$[d]	$HC(OEt)_3$	$C_6H_5CH{=}NOCH(OEt)_2$	52540-30-4	114-115 (0.8)	86
$C_6H_5CH{=}NOH$	$CH_3C(OEt)_3$	$C_6H_5CH{=}NOCCH_3(OEt)_2$	52540-31-5	100 (0.6)	88
p-$ClC_6H_4CH{=}NOH$[e]	$HC(OEt)_3$	p-$ClC_6H_4CH{=}NOCH(OEt)_2$	52540-32-6	135 (1.0)	89
p-$ClC_6H_4CH{=}NOH$	$CH_3C(OEt)_3$	p-$ClC_6H_4CH{=}NOCCH_3(OEt)_2$	52540-33-7	135 (0.8)	86
p-$CH_3OC_6H_4CH{=}NOH$	$HC(OEt)_3$	p-$CH_3OC_6H_4CH{=}NOCH(OEt)_2$	52540-34-8	144 (0.8)	84
p-$CH_3OC_6H_4CH{=}NOH$	$CH_3C(OEt)_3$	p-$CH_3OC_6H_4CH{=}NOCCH_3(OEt)_2$	52540-35-9	170 (0.8)	66

[a] The ratio oxime:ortho ester was in all examples 1:1 (usually a 100-mmol scale). [b] Analytical and all spectral data are in agreement with the structures. [c] Yields are of the isolated products. [d] Registry no. 622-32-2 (*Z*). [e] 3848-36-0. [f] 1663-61-2.

can be carried out neat by removing the alcohol and the ester from the reaction mixture as they are formed, or in solution. For example, the fragmentation of *n*-butyraldehyde oxime diethyl orthoacetate as a 1 *M* solution in toluene, ether, tetrahydrofuran, nitromethane, or dimethyl sulfoxide, in the presence of 2 mol % of methanesulfonic acid at room temperature was complete in less than 30 min.[6] The reaction in ethyl acetate or in acetone was much slower, requiring more than 3 hr for completion at 77° and 56°, respectively. On the other hand, the same transformation in sulfur dioxide solution in the absence of acid catalyst required only several minutes at room temperature. The nmr analysis indicated that the derivative of the *Z* oxime reacted somewhat slower than the *E* isomer.

The fragmentation reaction of benzaldehyde oxime diethyl ortho ester was considerably slower even in sulfur dioxide solution. For example, the corresponding orthoformate and orthoacetate required 48 hr at room temperature to give 90 and 60% yields of benzonitrile, respectively.[7,8]

We also found that ortho esters are very effective reagents for Beckmann fragmentation of α-oximino ketones and α-oximino ketone acetals (eq 4 and 5). Heating a mixture of commercial benzil monooxime with 10% excess of triethyl orthoformate in liquid sulfur dioxide at 75° gave an almost quantitative yield of ethyl benzoate and benzonitrile. Similarly, a solution of 2-oximinocyclohexanone dimethyl ketal and trimethyl orthoformate in sulfur dioxide



containing catalytic amounts of methanesulfonic acid, after short reflux at −10°, afforded a quantitative yield of 5-cyanopentanoic acid methyl ester. These reactions presumably involve intermediate formation of the corresponding oxime ortho esters which undergo the indicated Beckmann fragmentation *in situ*.

Experimental Section

The oximes and ortho esters used in this work were commercial samples purified when necessary by either distillation or crystallization. Boiling and melting points of the products are uncorrected. Glpc analyses were carried out generally on a Hewlett-Packard 5700A instrument using 3- or 6-ft columns of 10% SE-30 on Chromosorb W. Proton nmr spectra were recorded on either Varian A-60 MHz or HA-100 MHz instruments.

Typical Procedure for the Conversion of Oximes to Nitriles. A mixture of *n*-heptaldehyde oxime (15.0 ml, 100 mmol) and triethyl orthoformate (20.0 ml, 120 mmol), containing a drop

of methanesulfonic acid, was placed in a flask attached to a short distilling column and heated to distil the ethyl formate and ethanol formed in the reaction. When the distillation of the ester and alcohol ceased, the residue was distilled *in vacuo* to give a 95% yield of *n*-heptanoic acid nitrile, bp 70–72° (10 mm).

Typical Procedure for Conversion of Oxime Diethyl Ortho Esters. A. In Chloroform.[9] A solution of *n*-butyraldehyde oxime diethyl orthoacetate (20.3 g, 100 mmol) in chloroform (100 ml) was placed in a flask attached to a short distilling column and 0.2 g of methanesulfonic acid added. The solution was stirred at room temperature and the extent of the reaction was followed by neutralizing small aliquots which were then analyzed by glpc. After 30 min the reaction was complete and distillation afforded 5.48 g (94% yield) of *n*-butyronitrile.

B. In Sulfur Dioxide. A 100-ml heavy glass ampoule containing benzaldehyde oxime diethyl orthoformate (19.5 g, 100 mmol) was charged with approximately 50 ml of liquid sulfur dioxide at −70°, sealed, and kept at room temperature[10] for 48 hr. The ampoule was cooled in Dry Ice, opened, and the content transferred into a distilling flask containing 100 ml of cold chloroform. The flask was attached to a short column and excess of solvents removed *in vacuo*. The residue was distilled to give 9.3 g (90%) of benzonitrile, bp 68–70° (10 mm).

Typical Procedure for Preparation of Oxime Diethyl Ortho Esters. A solution of *n*-butyraldehyde oxime (a mixture of *Z* and *E* isomers in approximate ratio of 3:2) (8.7 g, 100 mmol) and triethyl orthoacetate (16.2 g, 100 mmol) was placed in a distilling flask attached to a short distilling column and heated at 120–150° until 4.6 g of ethanol distilled. Vacuum distillation of the residue gave a 95% yield of *n*-butyraldehyde oxime diethyl orthoacetate: bp 47–49° (0.6 mm); nmr ($CDCl_3$) δ 7.38 (t) and 6.68 (t) [1 H (total)], 3.54 (q, 4 H), 2.21 (m, 2 H), 1.5 (m + s, 5 H), 1.21 (t, 9 H).

Anal. Calcd for $C_{10}H_{21}NO_3$: C, 59.08; H, 10.41; N, 6.92. Found: C, 59.30; H, 10.50; N, 6.81.

Beckmann Fragmentation of Benzil Monooxime. A 50-ml heavy glass ampoule containing benzil monooxime (2.25 g, 10 mmol) and triethyl orthoformate (1.52 g, 11 mmol) was charged with approximately 25 ml of liquid sulfur dioxide at −70°, sealed, and heated at 72° for 70 hr. The ampoule was placed in Dry Ice, opened, and the contents were diluted with chloroform. Glpc analysis indicated a 95 and 98% yield of benzonitrile and ethyl benzoate, respectively.

Beckmann Fragmentation of 2-Oximinocyclohexanone Dimethyl Ketal. A 50-ml three-neck flask equipped with a magnetic stirrer, a Dry Ice condenser, and a nitrogen bubbler was charged with approximately 20 ml of sulfur dioxide at −70°, and 2-oximinocyclohexanone dimethyl ketal (1.73 g, 10 mmol), trimethyl orthoformate (1.2 g, 11 mmol), and a drop of methanesulfonic acid were then added. The solution was maintained under reflux (~−10°) for 30 min, and then 10 ml of chloroform and an internal standard were added. Glpc analysis indicated a 97% yield of 5-cyanopentanoic acid methyl ester.

Registry No.—Benzil monooxime, 14090-77-8; 2-oximinocyclohexanone dimethyl ketal, 52540-36-0.

References and Notes

(1) Mukaiyama, *et al.*, have reported[2] the synthesis of several oxime dialkyl ortho esters by the addition of an oxime to a ketene acetal.
(2) T. Mukaiyama, K. Tonooka, and K. Inoue, *J. Org. Chem.*, **26**, 2202 (1961).
(3) This observation is in accord with the known tendency of isomeric oximes to undergo various acylation reactions without isomerization.[4]
(4) P. A. S. Smith, "Open Chain Nitrogen Compounds," Vol. 2, W. A. Benjamin, New York, N.Y., 1966, p 46.
(5) We have also shown that oximes react with various ketone acetals to give the corresponding oxime alkyl ketone acetals. These, in turn, also undergo similar transformations as the oxime dialkyl ortho esters.
(6) The extent of the reaction was followed either by nmr or glpc analysis or both.
(7) This slow transformation is very likely a consequence of the syn arrangement of the carbon–hydrogen and nitrogen–oxygen bonds in the benzaldehyde oxime ortho ester, which eventually undergoes rate-determining isomerization to the more reactive anti isomer under the reaction conditions.
(8) Nmr analysis indicated that these oxime ortho esters were undergoing a series of reversible disproportionation reactions which will be discussed elsewhere.
(9) The experimental procedure in other solvents, *e.g.*, toluene, ether, tetrahydrofuran, or nitromethane, was essentially the same.
(10) When the reaction was unusually slow, as in the case of *p*-methoxybenzaldehyde derivatives, the ampoule was heated at 70°, cooled in Dry Ice, opened, and worked up in the same way.

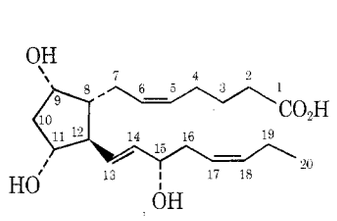
Synthesis and Resolution of 2-Hydroxyheptanoic Acid

Frank M. Hauser,* M. L. Coleman, R. C. Huffman, and F. I. Carroll

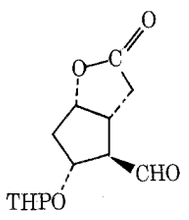
Chemistry and Life Sciences Division, Research Triangle Institute, Post Office Box 12194, Research Triangle Park, North Carolina 27709

Received February 8, 1974

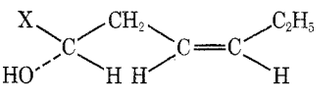
In an elegant synthesis of PGE_3 (**1**) reported by Corey and coworkers[1] the (*S*)-15-hydroxy-13,17-octadienyl side chain at C-12 was introduced stereospecifically by the condensation of the optically active aldehyde (**2**) with the ylide derived from the phosphonium salt (**3c**). The salt (**3c**) was prepared from (*S*)-(−)-malic acid by the multistep process (*S*)-(−)-malic acid → **3a** → **3b** → **3c**. It is apparent that the (*S*)-15-hydroxy-13-octenyl side chain at C-12 of prostaglandins and prostaglandin analogs could be introduced by the condensation of the ylide derived from the phosphonium salt (**5c**) with the appropriate aldehyde. The salt (**5c**) could be prepared from (+)-2-hydroxyheptanoic acid [(+)-**4**] by the sequence (+)-**4** → **5a** → **5b** → **5c**. In the present paper, we present an efficient and economical synthesis and resolution of (±)-2-hydroxyheptanoic acid [(±)-**4**].



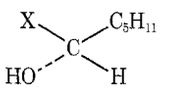
1



2



3a, X = CH_2OH
b, X = CH_2I
c, X = $CH_2\overset{+}{P}(C_6H_5)_3$



4, X = CO_2H
5a, X = CH_2OH
b, X = CH_2I
c, X = $CH_2\overset{+}{P}(C_6H_5)_3$

The preparation of (±)-**4** was accomplished in excellent yield from the cyanohydrin of hexanal. Numerous salts of (±)-2-hydroxyheptanoic acid with optically active amines were prepared, but only two, quinine and dehydroabeitylamine, were sufficiently crystalline to warrant further investigation. Recrystallization of both salts and liberation of the acid gave optically active material with opposite rotations. A large scale resolution employing quinine was performed and, in the very early recrystallizations of the quinine salt, two types of crystals were apparent. The progress of the resolution was followed by obtaining the rotation of the liberated and recrystallized acid. When a value of $[\alpha]^{25}D$ +5.55° was obtained, further recrystallization of the quinine salt did not increase the rotation of the acid.

The initial filtrate from the resolution of the plus (+) isomer was acidified, and the enriched minus (−) isomer was converted to its dehydroabeitylamine salt. The progress of the resolution of this isomer was followed as before with the final acid rotation being $[\alpha]^{26}D$ −5.52°.

In a study which established the absolute configuration of the C-15 hydroxyl group of prostaglandins, Nugteren and coworkers[2] degraded certain prostaglandins by ozonolysis and obtained 2-hydroxyheptanoic acid which possessed an optical rotation of $[\alpha]^{25}D$ +6.9, *c* 5.8, $CHCl_3$. Since the magnitude of our final rotations is different from