

Nylon-9 from Unsaturated Fatty Derivatives: Preparation and Characterization

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ABSTRACT

Methyl azelaaldehyde, soy nitriles, and oleonitrile were compared as starting materials for making nylon-9. Less difficulty was encountered in purifying intermediates from oleonitrile than from the other two. All 3 routes involved hydrolysis of methyl 9-aminononanoate to 9-aminononanoic acid. Self catalyzed hydrolysis of the amino ester in water produced low yields of monomer owing to oligomer formation, but hydrolysis in the presence of barium hydroxide was more successful. Polymerization of the amino acid proceeded smoothly with 9-acetamidononanoic acid used to control mol wt. Strength and moisture absorbing properties were determined for nylon-9, as well as for nylon-6/9, -9/11, and -9/12 copolymers. Nylon-9 has attractive properties, offering tensile yield and flexural strengths approaching those of nylon-6, while having low water absorption closely approaching that of nylon-11 and -12. The cost of producing 10,000,000 lb/year of nylon-9 from purchased oleonitrile was estimated to be \$0.99/lb at 100% yield and \$2.18/lb at 35.5% yield. Further process development is needed before yields in a commercial plant can be forecast.

INTRODUCTION

This work was undertaken to evaluate commercially significant properties of nylon-9, and to assess the practicality of selected processes for its preparation from unsaturated fatty products. Such starting materials are now receiving attention as potential alternates to petrochemicals derived from irreplaceable fossil oils. At present, nylon-11 from castor oil (1) and azelaic acid from oleic acid (2) are well known examples of how the plastics industry uses unsaturated fatty products; nylon-13 (3) and -13,13 (4) from crambe oil and their properties (5) have already been described.

The majority of vegetable oils contain fatty acids unsaturated in the 9,10-position, so that ozonolytic cleavage of the chain produces a 9-carbon bifunctional and a 9-carbon monofunctional product, e.g., azelaic and pelargonic acids. A good potential source for such unsaturated fatty derivatives is soybean oil, which is readily available. Crambe oil has unusually large amounts of erucic acid, a 22-carbon unsaturated acid, with unsaturation in the 13,14-position.

9-Aminononanoic acid can be prepared from soybean oil through methyl 9-aminononanoate (6) or 9-aminononamide (7). The first method involves alcoholysis, reductive ozonolysis, reductive amination, and hydrolysis reactions, whereas, the second involves aminolysis in addition to the other reactions. Methyl 9-aminononanoate forms as the consequence of working with intermediates as methyl esters so they can be isolated and purified by fractional distillation; other simple alkyl esters can also be used (8). The amino ester requires hydrolysis to the amino acid because amino esters do not produce as high mol wt products as the amino acid (9). When isolated, the amino ester is unstable and tends to condense to oligomers even at room temperature, although it is stable in an alcoholic solution (6).

An alternate route to the amino acid is through oleo-

nitrile, involving oxidative ozonolysis and hydrogenation (10). We investigated and compared methyl soyate, soy nitriles, and oleonitriles as starting materials for nylon-9 synthesis.

EXPERIMENTAL PROCEDURES

Methyl Soyate

Refined, bleached soybean oil was subjected to potassium hydroxide catalyzed methanolysis according to the procedure of Throckmorton, et al., (11). A fraction boiling between 139 and 175 C at 0.5-0.8 mm Hg was collected as methyl soyate (88% yield).

Methyl Azelaaldehyde

The methyl soyate was reductively ozonized according to the procedure of Throckmorton, et al., (11,12). Methyl soyate (400 g) was mixed with 150 ml water, and an ozone-oxygen mixture containing 37 mg ozone/liter was passed through the water-in-oil emulsion at the rate of 7 liters/min for 5 hr. The mixture was transferred to a hydrogenation autoclave (rocker type), palladium black catalyst (0.0625% on methyl soyate) was added, and hydrogenation was carried out at 500 psig at ca. 50 C for 3 hr. Temperatures higher than 50 C were avoided to prevent decomposition of the ozonolysis products. After cooling and settling overnight, the organic phase was separated, washed 3 times with 10% sodium bicarbonate and 3 times with water before drying. The product was distilled through a Widmer column (15 in. x 0.5 in.) at 0.1-0.3 mm Hg to collect a fraction boiling at 82-92 C (98% pure, 80% yield).

Oleonitrile

Oleic acid (6.75 lb of Hercules, Inc., Pamolyn 100, Wilmington, DE) and urea (1.98 lb) were charged to a 5-liter, 3-necked flask equipped with heating mantle, paddle stirrer, thermometer, and temperature controller (13). The mixture was heated and stirred under a nitrogen atmosphere to 295 ± 5 C and held at this temperature for 3 hr. The crude product assayed by gas chromatography contained 95% oleonitrile. Also used was commercial oleonitrile having the composition: oleonitrile, 66.4%; linoleonitrile, 10.0%; linolenitrile, 1.9%; palmitonitrile, 14.2%; myristonitrile, 6.0%; and stearonitrile, 1.7%.

Methyl 8-Cyanoctanoate

The oxidative ozonolysis of the crude oleonitrile was carried out in a 5-gal glass kettle equipped with a 4-in. diameter fan blade stirrer located near the bottom of the reactor, a gas inlet tube with the tip of the tube near the stirrer blade, a cooling-heating coil and a water cooled condenser. The reactor was charged with 10 lb oleonitrile, 9 lb glacial acetic acid, and 1 lb water. An ozone-oxygen gas stream containing ca. 1.8% ozone was passed into the reactor at the rate of 6.3 cfm for 4 hr, while the temperature of the reactants was maintained at 10 ± 2 C by passing water through the coil in the reactor. The ozonolysis product then was heated to 75 ± 2 C, and oxygen was passed into the reactor at the rate of 2.5 cfm. During the first 30 min when the reaction was exothermic, the temperature was controlled at 100 ± 2 C by water flowing through the reactor coil. After 30 min, the temperature was held at 100 ± 2 C by passing steam into the reactor coil as required.

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Oxidation was continued for a total of 2 hr; then the acetic acid was distilled out of the mixture, and the mixed acid product was left.

The mixed acid product was esterified with methanol in a 50-gal steam jacketed, stainless steel autoclave. The vessel was charged with 5 lb mixed acid product, 10.3 gal methanol, 12.3 gal 1,2-dichloroethane, and 1.6 lb sulfuric acid. This mixture was refluxed for 6 hr with stirring. After cooling, the reactor contents were washed twice with 4-gal portions of 5% sodium bicarbonate solution and twice with 10-gal portions of water. The 1,2-dichloroethane was removed by distillation, and the crude ester mixture recovered.

To recover methyl 8-cyanoctanoate, the crude ester mixture was vacuum distilled in an 8-gal stainless steel fractionating unit having a column 3 in. in diameter, 36 in. long, and filled with 1/4 in. protruded metal packing (Scientific Development Company, State College, PA). The distillation was carried out at 2.0 mm with a boil up rate of 6-8 liters/hr and a reflux ratio of 1:1. Fractions collected at ca. 5 C still-top temperature intervals were analyzed for methyl 8-cyanoctanoate by gas chromatography. Fractions assaying 90% or better were segregated in 73% yield for hydrogenation.

Methyl 9-Aminononanoate

Two methods were used successfully: (a) methyl azelaaldehyde (131 g) was charged to a rocking autoclave (American Instrument Co., Silver Spring, MD), together with 10 g nickel-on-kieselguhr catalyst (Girdler G49A), 640 ml methanol saturated with ammonia and 50 ml conc. ammonium hydroxide. Hydrogenation was carried out at 1500 psig and 85 C for 3 hr; (b) methyl 8-cyanoctanoate (3.5 kg), dissolved in 9.2 liters methanol containing 180 g Raney nickel catalyst, and 1.6 kg ammonia were charged to a 5-gal stirred autoclave (Autoclave Engineers, Erie, PA). Hydrogenation was carried out at 125 C and 700 psig hydrogen pressure for 3 hr. The contents of the autoclave then were cooled to ca. 50 C and filtered.

9-Aminononanoic Acid

The alcoholic solution of the amino ester was treated with and without barium hydroxide, used to prevent oligomer formation.

Without barium hydroxide. The filtered product from the hydrogenation of methyl 8-cyanoctanoate as described above was added slowly (ca. 4 hr) to 18 gal hot water (ca. 90 C) in a stirred autoclave. After methanol was distilled, the mixture was held under reflux for 24 hr, and then water was distilled to reduce the volume to ca. 10 gal. This solution was passed through a bed of granular activated charcoal and a cartridge filter. The charcoal treated and filtered solution was held at about 50 C for 2 hr before filtering to remove a waxy product that had formed. The filtered solution was cooled to room temperature, and an equal volume of acetone was added to it. After 24 hr, the first crop of 9-aminononanoic acid crystals was filtered off. The filtrate was cooled to ca. 2 C and held at this temperature for 24 hr, during which time a second crop of crystals formed. These were separated by filtration, combined with the first crop, and vacuum dried.

The crude 9-aminononanoic acid was further purified by dissolving the amino acid in hot water to form a 10% solution, stirring activated charcoal (1% Norite EX) and filter aid (Celite) into the solution, and filtering the solution hot. The filtered solution was held at 45-50 C for 2-4 hr, and then filtered to remove the waxy byproduct. An equal volume of acetone was added, and 2 crops of 9-aminononanoic acid crystals were collected as before. The total wt of amino acid recovered was 630 g for an overall yield of 19% from methyl 8-cyanoctanoate.

With barium hydroxide. The filtered crude amino ester

(250 g), 600 g water, and 140 g barium hydroxide octahydrate were added to a 500-ml, 3-necked flask, equipped with a stirrer, a distilling side arm, a condenser, and a thermometer, and heated to 100 C over 4 hr. During heating, methanol distilled over; toward the end, a small amount of other organic material distilled over. The temperature then was lowered to 85-90 C and held there for 2 hr, after which time 20% sulfuric acid was added to bring the pH of this mixture to 6.0-6.5. The mixture was held at 85-90 C for 4 hr, and then filtered in a steam heated Buchner funnel through a Whatman No. 42 filter paper topped with a 1/4-in. thick mat of Celite. The filter was then washed with hot water.

Although some 9-aminononanoic acid crystallized as the filtrate cooled, it was not separated. An equal volume of the acetone was added to the filtrate, and after ca. 24 hr, the amino acid was filtered off. The filtrate was stored in an ice bath overnight, and a second crop of 9-aminononanoic acid crystals was recovered and combined with the first. The total wt of amino acid recovered was 165 g for an overall yield of 72% from methyl 8-cyanoctanoate.

Nylon-9

A 500-ml, round bottomed flask, which had been lightly coated inside with a silicone release agent (DC808, Dow Corning Corp., Midland, MI) to prevent adhesion of the nylon to the flask, was charged with 115.8 g 9-aminononanoic acid and 0.765 g 9-acetamidononanoic acid (14). The flask was flushed with nitrogen to remove air, immersed in an oil bath at 240 C, and evacuated with a water aspirator. After 1 hr the aspirator was replaced by an oil pump and the pressure was reduced to ca. 0.5 mm. After 2 hr the heat was removed and polymer was cooled under nitrogen.

RESULTS AND DISCUSSION

Synthesis of 9-Aminononanoic Acid from Soybean Oil

Ozonolysis of methyl soyate and hydrogenation of the ozonolysis products to methyl azelaaldehyde presented no operational problems. Water was satisfactory as the ozonolysis medium, and did not require removal from the ozone before hydrogenation. When methanol was substituted for water as the ozonolysis medium, yields were lower. Hydrogenation of the ozonolysis products to methyl azelaaldehyde in the presence of palladium black catalyst was simple and straight forward, requiring moderate temperature and pressure. Therefore, no variations of hydrogenation conditions were tried.

Methyl soyate was converted to methyl azelaaldehyde in 86-93% yield. Separation of a pure (> 95%) methyl azelaaldehyde by fractional distillation was difficult because an unidentified impurity had a bp close to that of the desired product. Recovered yields of methyl azelaaldehyde were routinely 65-70%, and occasionally 80% of theory. Yields could probably be improved by distilling more efficiently or by using a higher alkyl soyate that gives a greater difference in bp between alkyl azelaaldehyde and the impurity.

Conversion of methyl azelaaldehyde to methyl 9-aminononanoate in the presence of nickel-on-kieselguhr catalyst or Raney nickel catalyst caused no operational problems. Methyl 9-aminononanoate was not isolated because of its facile self condensation, but was hydrolyzed immediately to the amino acid by adding the methanolic solution from the hydrogenation reactor slowly to boiling water. The aqueous solution of 9-aminononanoic acid was filtered at ca. 50 C to remove a waxy byproduct, and then was evaporated to dryness to recover the amino acid. On this basis, the yield of 9-aminononanoic acid from methyl azelaaldehyde was ca. 70% of theory. The wide mp range

(165-175 C) of amino acid recovered in this way indicated an impure product.

Synthesis of 9-Aminononanoic Acid from Oleic Acid

Oleic acid was converted easily to oleonitrile by the procedure of Kaplan (13). About 95 lb of oleonitrile was made in 15 preparations and the crude products assayed by gas chromatography contained 92-95% oleonitrile. Simple distillation of the crude product at 2-3 mm gave a refined oleonitrile (98+%) in ca. 75% yield. Subsequently, the crude product proved as satisfactory as the refined product in the synthesis of 9-aminononanoic acid.

The oxidative-ozonolysis procedure was conventional. Nine of these reactions were carried out with 10-lb charges of oleonitrile without difficulty. Esterification of mixed acids (primarily 8-cyanooctanoic and nonanoic acids), and distillation of the resulting esters involved no elements of novelty. Distillation of the methyl esters of the mixed acids was carried out at a low reflux ratio (1:1) to minimize distillation time. There was an appreciable loss of methyl 8-cyanooctanoate in discarded distillate fractions. With the experimental procedures used, the recovered yield of methyl 8-cyanooctanoate averaged 74% of the theory based on oleonitrile charged. It is estimated that the yield could be raised to at least 85% of theory by more efficient fractionation.

The nitrile route was also attempted with soy nitriles. However, yields were lower in the oxidative ozonolysis step, and the overall process was not as satisfactory as that starting with oleonitrile.

Recovery of 9-Aminononanoic Acid

Recovering 9-aminononanoic acid created problems. During hydrolysis of the methyl ester in hot water, a waxy byproduct of undetermined composition formed and was difficult to separate. The amino acid crystallized from water or water-acetone mixtures as fine hydrophilic crystals, which contained the mother liquor. Consequently, the crude amino acid had to be recrystallized (often twice) to obtain a product suitable for polymerization. Recovered yields in the conversion of methyl 9-aminononanoate to polymer grade 9-aminononanoic acid by this method were, at best, 50% of theory, and generally appreciably lower. The low yields from recrystallization point out the need for further studies on crystal and solubility properties of the amino acid. With such data in hand, much improved recoveries can be expected.

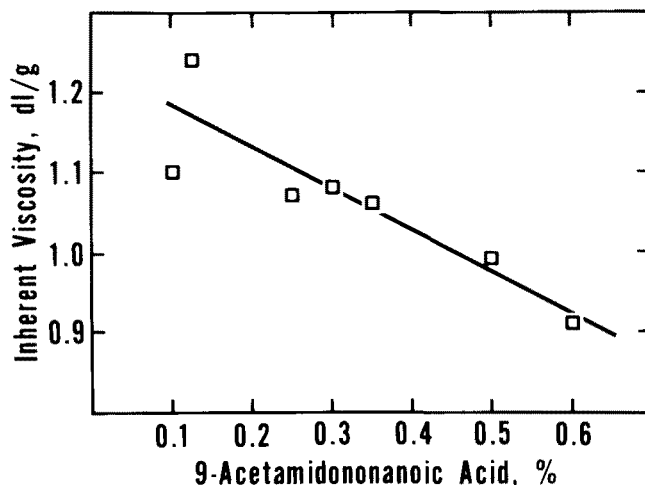


FIG. 1. Effect of chain terminator on inherent viscosity.

Even the limited work with barium hydroxide to cleave the ester promised an improved recovery of 9-aminononanoic acid. In a single experiment, the recovered yield of 9-aminononanoic acid having a melting point of 180-182 C and suitable for polymerization was 72% of theory.

Polymerization of 9-Aminononanoic Acid

9-Aminononanoic acid polymerized to nylon-9 without difficulty. The mol wt of nylon-9 was controlled by using 9-acetamidononanoic acid as a chain terminator. The relationship between inherent viscosity of nylon-9 and concentration of terminator is shown in Figure 1. Variations in purity of the 9-aminononanoic acid can shift the position of the curve.

Properties of Nylon-9

Of course, tensile properties and melt viscosity of nylon-9 are influenced by mol wt (Table I). Tensile tests were made with monofilaments prepared with a small ram extruder and with ones oriented by cold drawing. Tensile strength was changed little in the range of inherent viscosities from 1.03 to 1.28 dl/g, but melt viscosity increased substantially. Thus, the choice of polymer in this viscosity range depends on the melt viscosity required for fabrication rather than on tensile properties. Tensile factors (15),

TABLE I

Inherent Viscosity, Melt Viscosity, and Tensile Properties of Nylon-9 Monofilaments

Property	ASTM method ^a	Sample			
		A	B	C	D
Inherent viscosity, dl/g	D-2857 ^b	0.77	0.84	1.03	1.28
Melt viscosity, poises at temperature (C)	D-1238	730 225	1170 225	1560 245	6840 265
Tensile properties	D-2256				
Undrawn					
Tensile strength (psi)		16,400	16,600	20,500	22,000
Tenacity (g/d)		1.1	1.2	1.5	1.6
Initial modulus (g/d)		22	18	20	27
Elongation at break (%)		340	470	420	390
Tensile factor ^c		20	26	32	32
Cold drawn					
Tenacity (g/d)		4.0	3.8	5.0	5.2
Initial modulus (g/d)		28	28	30	31
Elongation at break (%)		29	43	38	38
Tensile factor ^c		22	25	32	32

^aASTM = American Society for Testing and Materials, Philadelphia, PA.

^b0.5 g/dl in hexafluoroisopropanol at 30 C.

^cProduct of tenacity and the square root of elongation at break (15).

TABLE II
Physical Properties of Nylons-9, -11, and -12

Property	ASTM ^a method	Nylon-9 ^b	Nylon-11	Nylon-12
Crystalline mp (C) ^c		211	198	185
Specific gravity	D-792	1.082	1.050	1.020
Hardness (Shore D)	D-2240	80	77	75
Deflection temperature at 264 psi (C)	D-648	56	54	57
Yield strength (psi)	D-1708	6400	5300	4900
Elongation at yield (%)	D-1708	24	26	11
Tensile strength (psi)	D-1708	6800	5300	5500
Tensile strength at break (psi)	D-1708	6300	4600	5400
Elongation at break (%)	D-1708	180	100	250
Compression stress at 10% (psi)	D-695	6100	5400	5600
Compression modulus (psi)	D-695	68,000	63,000	64,000
Compression stress at yield (psi)	D-695	4900	4400	4500
Compression at yield (%)	D-695	7	6	7
Flexural strength (psi)	D-790	10,300	8700	8700
Flexural modulus (psi)	D-790	190,000	155,000	159,000
Water absorption, 24 hr at 23 C (%)	D-570	0.35	0.29	0.27
Water absorption, 7 days at 23 C (%)	D-570	0.77	0.62	0.58

^aASTM = American Society for Testing and Materials, Philadelphia, PA.

^bAverage of 2 polymers.

^cBy differential thermal analysis.

which are indicative of toughness and which are more sensitive to changes in mol wt, increase progressively as inherent viscosity of nylon-9 increases from 0.77 to 1.03 dl/g, but no further increase occurs to 1.28 dl/g. A nylon-9 having an inherent viscosity of ca. 1.0 dl/g seems to offer the best compromise of tensile properties and processability.

Properties of nylon-9 and those of commercial nylon-11 and nylon-12 resins are compared in Table II. Physical property measurements of 2 nylon-9 samples, differing only slightly in inherent viscosity are averaged.

As would be expected, nylon-9 has somewhat higher tensile strength, yield strength, and flexural strength than nylon-11 and nylon-12. Nylon-9 is more rigid than nylon-11 and -12, as evidenced by differences in flexural modulus. The water absorption characteristics of nylon-9 are interesting and pertinent. Nylon-9 absorbs only slightly more water in the ASTM D-570 test than does nylon-11 or nylon-12, and, thus, provides similar low water absorption characteristic of nylon-11 and -12 while offering higher strength and rigidity.

Reinforced nylon-9 was prepared with glass fibers and with glass beads as the reinforcement at 20% concentration (Table III). Glass fiber reinforcement increased heat distortion temperature materially, increased flexural strength and modulus modestly, reduced impact strength somewhat, and reduced elongation at break sharply. Glass bead reinforcement offered no important beneficial effects.

Copolymers were prepared by heating equal wt of nylon-9 with other nylons at 290 C under nitrogen until the differential thermal analysis of samples taken from the mixture showed only a single endotherm peak. Selected proper-

TABLE III
Properties of Glass Reinforced Nylon-9

Properties	Reinforcement			None
	ASTM method ^a	Glass fiber ^b	Glass beads ^c	
Deflection temperature at 264 psi (C)	D-648	170	56	54
Hardness (Shore D)	D-2240	80	80	80
Flexural strength (psi)	D-790	13,900	10,900	10,700
Flexural modulus (psi)	D-790	286,000	225,000	185,000
Tensile strength (psi)	D-1708	7200	5600	6900
Elongation at break (%)	D-1708	15	32	200

^aASTM = American Society for Testing and Materials, Philadelphia, PA.

^b20% Owens Corning 411AA chopped fibers.

^c20% Potters Brothers No. 70-140 glass beads.

ties of copolymers made with nylons-6, -11, and -12 are given in Table IV. The combination of nylon-6 and nylon-9 is noteworthy, because properties generally are not greatly different from those of nylon-9 alone, including a water absorption of 1.35% compared to 0.77% for nylon-9 and about 10% for nylon-6. Slight variations in test data for nylon-9 reflect different polymer preparations.

Estimated Costs

In early 1973 estimates were calculated for capital investment and operating costs for a plant with a capacity of 10 million lb/year (Table V). The estimates were based

TABLE IV
Properties of Nylon Copolymers^a

Properties	ASTM ^b method	Nylon			
		9	6/9	9/11	9/12
Crystalline melting point (C) ^c		211	178	160	168
Hardness (Shore D)	D-2240	80	75	67	70
Tensile strength (psi)	D-1708	6800	6300	5000	5300
Elongation at break (%)	D-1708	180	280	240	240
Torsional modulus (psi)	D-1043	--	51,000	--	54,000
Water absorption, 7 days at 23 C (%)	D-570	0.77	1.35	0.88	0.58

^a1:1 wt ratio.

^bASTM: American Society for Testing and Materials, Philadelphia, PA.

^cBy differential thermal analysis.

TABLE V
Estimated Costs of Producing 10 Million Lb Nylon-9 Per Year^a

Item	Costs based on 35.5% yields ^b		Costs based on 100% yields ^c	
	\$/yr	\$/lb	\$/yr	\$/lb
Capital costs	\$11,083,000		\$9,210,000	
Operating costs				
Raw materials ^d	17,114,000	1.711	6,210,000	0.621
Utilities	800,000	0.880	700,000	0.070
Labor	460,000	0.046	460,000	0.046
Maintenance	665,000	0.067	552,000	0.055
Payroll extras	92,000	0.009	92,000	0.009
Amortization	1,578,000	0.158	1,320,000	0.132
Taxes and insurance	111,000	0.011	92,000	0.009
General and administrative	1,020,000	0.102	484,000	0.049
Total operating costs	21,840,000	2.184	9,910,000	0.991

^aCalculations were made in early 1973.

^b20.9 lb nylon-9 per 100 lb oleonitrile.

^c58.9 lb nylon-9 per 100 lb oleonitrile.

^dIncluding 47,600,00 and 16,900,00 lb, respectively, of oleonitrile at \$0.2975/lb.

on commercial oleonitrile as the starting material at a cost of \$0.2975/lb. Seven major processing steps were included: ozonolysis, esterification, distillation, hydrogenation, hydrolysis, purification, and polymerization.

A major factor influencing estimated cost is yield (Table V). It is quite evident that further process studies are needed to improve the overall yield of 35.5%, which results in a prohibitively high production cost of \$2.18/lb. Even at 100% yield, the cost is \$0.99/lb. However, no byproduct credit was taken, and sale of such byproducts as methyl pelargonate and methyl palmitate could significantly reduce the cost. About 2 lb of methyl pelargonate are produced for every pound of nylon-9 at the 35.5% yield. Although pelargonates are useful in plasticizers and synthetic ester lubricants, there might be problems associated with disposing of such a large amount (20 million lb/yr). At 100% yields, there would be about a 1:1 wt ratio for these products. Excluding raw materials, processing costs are \$0.47 and \$0.37/lb of nylon-9 at respective yields of 35.5% and 100%.

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ERRATUM

In the article by D.N. Grindley and S.A. El Sarrag entitled "The Oxidation of 9:10 Diketostearic Acid by Peracetic Acid (Baeyer & Villiger Reaction)" (*JAOCS* 49:338 (1972)), the reference by N.A. Khan and M.S. Newman was cited incorrectly. It should be *J. Org. Chem.* 17:1063 (1952).