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Nylon-1313 from Brassylic Acid

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Multi-pound production of nylon-1313 demonstrates that there are no serious obstacles to commercial production of this long-chain polyamide. The synthesis of nylon-1313 is remarkably simple and straightforward when compared to the reactions required to produce nylon-11 and -12. In many ways nylon-1313 is comparable to these other nylons, but it is lower melting, slightly less dense, and more hydrophobic than either of its counterparts. This engineering resin can be produced economically using brassylic acid derived from crambe or other high-erucic acid oils. Predicted yield in a 10 million lb/year plant facility is about 83 lb of molten polymer per 100 lb of crude brassylic acid, or the equivalent of about 96% of theory.

Processing advantages, durability, and possible energy conservation benefits (Fallwell, 1975) assure an expanding demand for engineering plastics, but to continue increasing production from petroleum or other nonrenewable resources may be unwise. In this context, agricultural oils are appealing alternatives as feedstocks for the plastics industry; although unlikely to supplant petroleum within the foreseeable future, these oils can already supply a major share of demand for long-chain engineering nylons, as does castor oil for nylon-11, an established industrial polymer. Two other commercial nylons, nylon-69 and nylon-610, are derivable, in theory, from seed oils. Similarly, nylon-9 has been prepared in the laboratory from soybean oil (Kohlhase et al., 1970; Miller et al., 1971; Perkins et al., 1975), and other diverse polyamides are theoretically feasible from the many less common oils of nature

provided such botanical sources can be improved agronomically and made practical commercially. Among prospective nylons from annual crops, those incorporating 13-carbon monomers are especially promising for domestic exploitation because the constituent monomers, brassylic (tridecanedioic) acid and 1,13-diaminotridecane, are easily prepared from erucic (*cis*-13-docosenoic) acid, a major constituent of certain rapeseed oils or of the new domestic high-erucic oilseed, i.e., crambe (Nieschlag and Wolff, 1971).

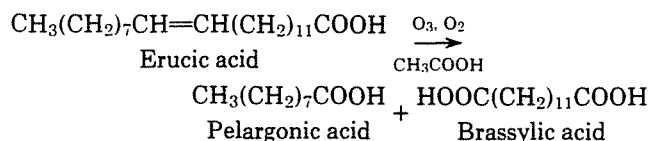
Important physical properties of nylon-1313 are listed in Table I. In many ways, nylon-1313 is comparable to nylons-11 and -12, but it is lower melting, slightly less dense, and more hydrophobic than either of its counterparts. These properties can be advantageous in specific applications. Lower density provides more bulk in molded articles per pound of resin, and

Table I. Typical Physical Properties of Nylon-1313

Specific gravity	1.01
Crystal melt temperature (DTA)	175–176 °C
Water absorption (immersed):	
After 72 h	0.28%
At equilibrium	0.75%
Moisture regain, 50% RH, 25 °C:	
After 10 days	0.18%
At equilibrium	0.27%
Tensile strength, psi	5210–5700
Tensile yield strength, psi	4640–4660
Elongation at break, %	130–290
Modulus of elasticity, psi	101 000–114 000
Flexural strength, psi	7850–10 200
Flexural modulus, psi	176 000–192 000
Hardness, shore D	72
Impact strength, ft lb/in.	2.6
Mold shrinkage in./in.	0.014
Coefficient of linear thermal expansion (in./in./ °C from –30 to +30 °C)	1.03×10^{-4}

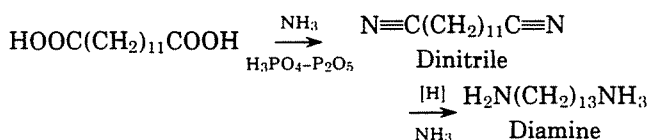
lower water absorption ensures dimensional stability in molded or extruded goods under extremes of humidity. Also, reduced processing temperatures, which are possible with this lower melting resin, may lower processing costs and help to avoid heat degradation.

In the preceding paper, Carlson et al. (1977) described the conversion of erucic acid to brassylic acid by oxidative ozonolysis.

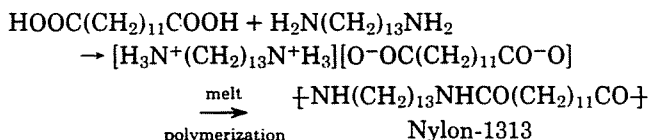


This versatile difunctional product has potential applications in plasticizers and lubricants analogous to those served by shorter chain dicarboxylic acids (Nieschlag et al., 1964; Critchley, 1962), but its use as a monomeric intermediate in nylon synthesis is of primary interest here.

Several different nylons can be made by reacting brassylic acid with various diamines: for example, with hexamethylenediamine to give nylon-613. To provide monomer for the companion polymer, nylon-136, or for nylon-1313, brassylic acid can be converted to a 13-carbon diamine, 1,13-diaminotridecane.



Combination of this diamine with brassylic acid yields a nylon salt, which is melt polymerized to nylon-1313.



The simplicity of this process, which is shown schematically in Figure 1, is quite striking when compared to reactions required to produce nylon-11 from castor oil (Genas, 1950) or nylon-12 from petrochemical feedstock (Griehl and Ruestem, 1970).

We intended no changes in this nylon-1313 process in scale-up because the synthesis had proven very dependable in the laboratory (Greene et al., 1967). Several improvements in reaction-control and materials-handling were necessary, however, and we were able to simplify the synthesis as de-

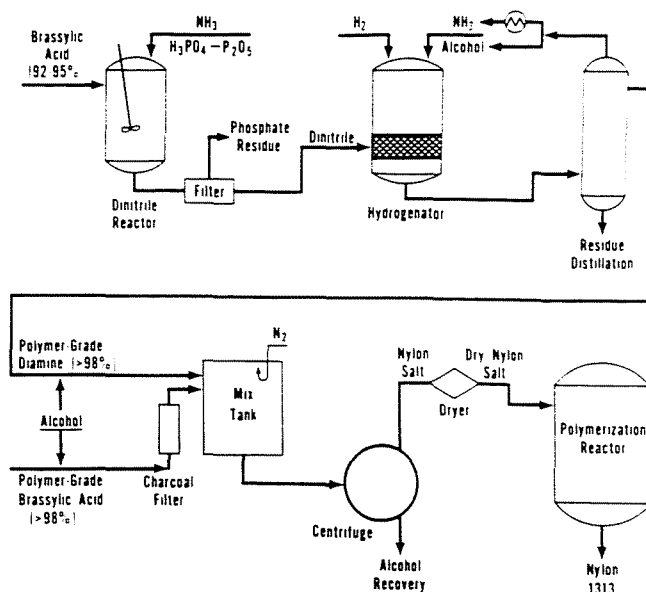


Figure 1. Process for making nylon 1313.

scribed in this report of the first preparation of nylon-1313 in multi-pound quantities.

Conversion of Brassylic Acid to Dinitrile

Crude brassylic acid (95% pure), as obtained from ozonolysis of erucic acid (Carlson et al., 1977), was used without purification for synthesis of the dinitrile, 1,11-dicyanoundecane. A 12-l. reaction vessel was charged with about 15 lb of brassylic acid and 0.15 lb of polyphosphoric acid. The mixture was melted under a nitrogen blanket; when the temperature reached about 150 °C, anhydrous ammonia was bubbled into the liquid at 4 l/min and the reaction temperature was increased to and held at 300 °C. This rather simple conversion required 18–24 h but involved little manpower. Any material blown from the reactor during the course of the reaction was collected in a trap and later combined with the contents of the reactor for processing. After the reaction was complete, the reactor contents were cooled slightly below room temperature and filtered to remove phosphate residues. In defense of such a crude process, we point out that yields were as high as 97% of theory and recovered product was 97–98% pure, as analyzed by GLC using a 1/8 in. × 12 ft column of 10% UC-W98 on 80–100 mesh Gas Chrom Q; column temperature, 200 °C; injection port and detector, 300 °C; helium flow, 50 ml/min.

A larger batch of dinitrile (ca. 24 lb), which was prepared using a motor-driven blade stirrer and more rapid ammoniation (8 l/min) through a fritted glass-tipped gas inlet tube, required only 8 h at 300 °C. A sample taken just as this reaction reached 300 °C assayed 68% dinitrile, which indicates that the reaction proceeds quite rapidly in the early stages and could yield reasonably high conversions in very short times. Presumably, technology for making other dinitriles, e.g., adiponitrile, via a pressurized continuous-flow process (Sittig, 1967) could be adapted to the commercial production of brassylonitrile.

Hydrogenation of Dinitrile

With cobalt-on-kieselguhr catalyst (G-67RS, Girdler Catalyst Division, Chemetron Corporation, Louisville, Ky.), experiments during scale-up proved that use of distilled dinitrile provided no chemical advantage over hydrogenation of dinitrile, as formed. Though we also found that distilled material hydrogenated faster, the opportunity to exclude this purification step from the original laboratory synthesis (Greene et al., 1967) offered a definite and overriding convenience for our

studies. Consequently, most of the diamine was made by the following procedure. A 5-gal autoclave (Autoclave Engineers, Inc., Erie, Pa.) was charged with 23.8 lb of crude dinitrile, 5.25 lb of methyl alcohol, and 0.48 lb of catalyst and then was purged three times with hydrogen by pressuring to 500 psi and bleeding to the atmosphere; a small charge of anhydrous ammonia (2.5 lb) was added to suppress secondary amine formation; and the autoclave was pressurized with hydrogen (1500 psi) and brought to temperature (120 °C). Hydrogenation was complete after 5–6 h; best yield of polymer-grade diamine was 86% of theory; average yield, 80%.

An alternate catalyst, No. 27 Raney active cobalt (Raney Catalyst Division, W. R. Grace Co., South Pittsburg, Tenn.), seemed to be poisoned by unknown constituents in the crude dinitrile, but distilled dinitrile was hydrogenated easily with this catalyst by the Armour Industrial Chemical Co. (McCook, Ill.). Approximately 20 lb of dinitrile in a stainless steel autoclave with 2% catalyst and a small ammonia charge was hydrogenated under 2000 psig hydrogen pressure at 120–145 °C for 6.5 h. The crude product was vacuum distilled (125–127 °C, 0.3 mm) through a 0.75 × 22 in. Vigreux column to give diamine of 97.7% purity in 98.5% yield from the dinitrile.

The hydrogenation product, after filtration to remove catalyst, was purified to polymer-grade diamine in an 8-gal stainless-steel distillation unit. This still was equipped with a 3-in. diameter column having a 36 in. long section packed with 1/4-in. protruded metal packing (Scientific Development Corp., P.O. Box 795, State College, Pa. 16801). Ammonia and methyl alcohol were distilled off at atmospheric pressure; the diamine was distilled at 1–2 mm pressure. Reflux ratio was 3:1, and the indicated boil-up rate was 6–7 lb/h. Fractions collected during distillation were analyzed for diamine content by GLC of solid samples using a 1/8 in. × 6 ft column of 3% OV-1 on 80–100 mesh Gas Chrom Q; column temperature, 150 °C; injection port and detector, 300 °C; helium flow, 50 ml/min. Those that assayed 98% or higher diamine were segregated as pure product; fractions less pure were combined and redistilled. The best recovery of diamine was 92% of that charged to the still pot. In this instance, the product was 99.0% pure.

Preparation of Nylon-1313 Salt

Nylon-1313 salt was formed by combining equimolar solutions of brassylic acid and 1,13-diaminotridecane in isopropyl alcohol. The nylon salt, which precipitated, was recovered by filtration, washed with isopropyl alcohol, and dried. Both monomers were of high purity before salt formation. The preparation of 99% pure diamine by distillation has been described above. The brassylic acid used here was crystallized from toluene after being passed through an activated charcoal column; it was over 98% pure (Carlson et al., 1977). This single recrystallization procedure allowed excellent recoveries and removed nearly as much color from crude brassylic acid as did a twofold crystallization and char treatment in ethanol that had been called for by the original laboratory procedure (Greene et al., 1967). In general, ethyl alcohol was avoided in these pilot studies because product recoveries were sometimes below expectations, and because of regulations concerning its use. Isopropyl alcohol was quite satisfactory as solvent for final charcoal treatment of brassylic acid and salt preparation, provided the alcohol was kept above 40 °C.

The following describes a typical nylon salt preparation. Thirty gallons of isopropyl alcohol was charged to a 55-gal drum fitted with a polyethylene liner. The alcohol was preheated to 40–50 °C with a steam coil. Twenty-one pounds of brassylic acid was added and the mixture was stirred with an air-driven stirrer (Model AR08, Lightnin Mixer, Mixing

Equipment Co., Inc., Rochester, N.Y.) that was fitted with two 3-in. diameter propeller blades mounted 2 in. and 10 in. above the center of the drum bottom. After the acid was dissolved, the solution was pumped (ca. 5 gal/h) through a 3 × 36 in. column containing 6 lb of a 4 × 14 granular activated charcoal (American Norit Co., Inc., Jacksonville, FL). Solution leaving the charcoal bed was collected under nitrogen in a tared and covered polyethylene-lined drum. Two weighed samples (5 ml each) were titrated with 3 N sodium hydroxide to assess the amount of brassylic acid present. The brassylic acid solution, heated to about 50 °C with a steam coil, was then stirred with the Lightnin Mixer operated at 2000–2500 rpm, while an equimolar quantity of diamine in 10 gal of isopropyl alcohol was mixed in at a rate of 3–3.5 gal/h. Stirring was continued for about 1 h after addition of the diamine. Exothermic salt formation raised the temperature to about 65 °C, but salt formed at this temperature was less solvated than that formed at lower temperatures. Under these conditions, the nylon-1313 salt precipitated as fine particles whose size, 0.5–1.5 μ in diameter, presented some operational problems with equipment at hand. The salt was best separated from alcohol in a Model AF 20-in. Tolhurst basket centrifuge (Ametek, Inc., East Moline, Ill.) lined with Type 2602 Netone filter paper (National Filter Media Corp., New Haven, Conn.). Salt slurry was poured into the operating centrifuge at about 5 gal/h; when 12–15 lb of salt had been loaded, the cake was centrifuged for 1 h and then washed three times with 1-gal portions of isopropyl alcohol. The washed cake was finally centrifuged for 2–3 h to reduce alcohol in the salt to below 15%. With an alcohol content below this level, the salt is a fine powder that can be handled easily; higher levels of alcohol cause pasty lumps.

Although suitable for a small-scale batch process, this method for isolating the salt is rather slow and does not remove all alcohol. Additional research would be necessary in this area to ensure faster and more complete separation in large-scale continuous production. Oddly enough, nylon-613 salt and nylon-136 salt precipitate as easy-to-handle particles. From all appearances, salt formation should be no problem in further development of these two related nylons.

The nylon-1313 salt was dried in stainless-steel trays (3/4 × 14 1/2 × 22 in.) in an electrically heated circulating oven with 1 1/2 in. space between trays. Care was taken that no lumps were larger than about 3/4 in. Nitrogen was flushed through the cool oven at a rate of 30 l/min for 2 h to sweep out residual oxygen. Then the nitrogen flow rate was adjusted to about 15 l/min while the temperature was raised to 65 °C over a period of about 3 h. Drying was continued for 8 h under a nitrogen flow of 8–10 l/min, and then the salt was allowed to cool to room temperature. Salt yields were essentially quantitative, but mechanical losses in drying and other handling amounted to about 5%.

Polymerization

In the original laboratory preparation of nylon-1313, polymer molecular weights were controlled by adding the diacetate salt of 1,13-diaminotridecane as a chain terminator (Greene et al., 1967). This is a sound idea based on the use of acetic acid as a terminator for nylon 66 (Sweeny, 1968) and affirmed in several small-scale experiments demonstrating that the molecular weights of nylon-1313 polymers could be predicted from terminator concentrations (Greene et al., 1967). Guided by such convincing evidence, we used the diacetate salt in our first large batch polymerization only to find that the product was an unwieldy mass. Much of this first preparation was of such high molecular weight that it had to be burned to remove it from the polymerization kettle. A bench-scale check via the original procedure confirmed the

Table II. Effect of Terminator on the Polymerization of Nylon-1313 Salt

Run	Dry salt charge, lb	Terminator		Molar ratio salt/terminator	Nylon-1313 discharge, lb	Inherent viscosity, dl/g
		Type	g/lb			
1	12.3	Diamine diacetate	1.40	236	7	...
2	15.2	Diamine diacetate	1.40	236	10	...
3	24.0	Diamine diacetate	1.75	189	12	1.2-1.4 ^a
4	11.6	Benzoic acid	1.02	118	7	0.87
5	13.3	Benzoic acid	1.02	118	12	0.81
6	22.8	Benzoic acid	1.02	118	19	1.01
7	21.3	Benzoic acid	1.02	118	18	1.08
8	15.8	Benzoic acid	1.50	80	16	1.09
9	25.5	Stearic acid	3.5	80	25	0.60
10	28.0	Stearic acid	3.5	80	27	0.74

^a Increase during pump out.

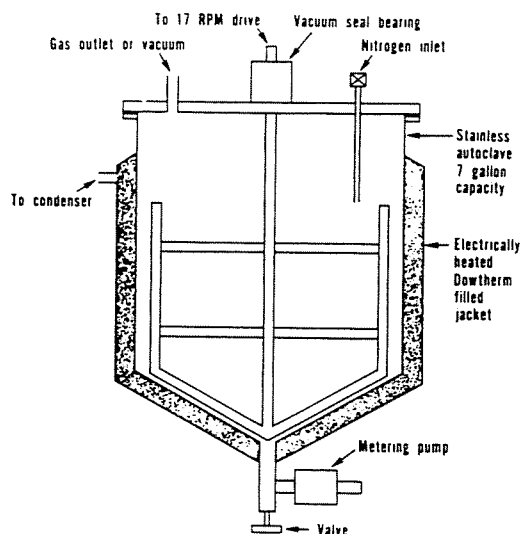


Figure 2. Polymerization autoclave.

method and demonstrated that all reactants were good quality, but a second pilot-scale polymerization also went beyond control even though terminator concentration had been increased by 50%. Subsequent studies established that stirring caused the troublesome over-polymerization by promoting loss of acetic acid from the molten resin. Less volatile acids, stearic, and to a lesser extent, benzoic, proved much more effective than 1,13-diaminotridecane diacetate at stabilizing the polymer molecular weight. These experiences are summarized in Table II.

The inherent viscosity of a preparation terminated with stearic acid (3.50 g/lb of dry nylon salt) was stable for 8 h at 220 °C, and a preparation terminated with benzoic acid (1.02 g/lb of salt) only increased from 0.81 dl/g to 0.89 dl/g during 6 h at 220 °C. Both stearic and benzoic acids were used successfully in different pilot-scale polymerizations, but the following procedure was preferred on the basis of all results from our studies. A polymerization autoclave (Figure 2) was charged with 28 lb of dry nylon 1313 salt, 98 g of stearic acid, and 63.5 g (0.5%) Ionox R330 antioxidant (Ethyl Corp., Baton Rouge, La.). The autoclave was evacuated and filled with nitrogen three times to purge oxygen, and nitrogen was then fed at 11 l/h. The stirrer was started to thoroughly mix the powder together, and heat was applied to the Dowtherm-filled jacket. The temperature of the autoclave contents reached 215 °C in 6 h and increased slowly to 225 °C over the next hour. Thereafter, the temperature of the polymer in the autoclave was held to about 225 °C for 2 h. The stirrer was stopped after 1 h at this temperature to allow entrapped gases to escape.

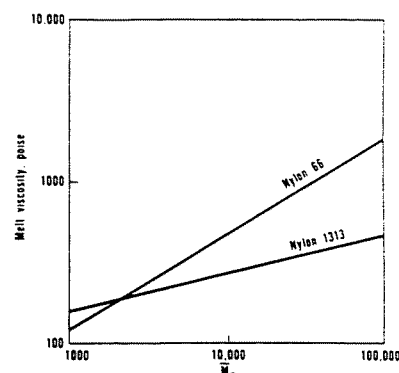


Figure 3. Melt viscosity vs. molecular weight for nylon-66 and nylon-1313. Data for nylon-66 from Sweeny and Zimmerman (1969).

Then over a period of about 45 min, the temperature of Dowtherm in the jacket was raised to 250 °C, the metering pump was heated to 240–245 °C, and molten polymer was pumped out at about 15 lb/h under 50 psig nitrogen pressure in the form of 1/8-in. monofilament, which was drawn through a 10 ft long water-filled quench tank. Upon leaving the tank, the monofilament passed through take-up rolls at about 54 ft/min and thence into a chopper, which converted the thick monofilament into 1/8 × 1/4 in. pellets.

Properties

Molecular size and shape are important structural features that have pronounced effects on the flow characteristics of polymers under shear-stress conditions (Billmeyer, 1971). Suitable terminators for nylon-1313, which allowed control of polymerization, also enabled comparisons that reflect the processability of the resin. As shown in Figure 3, which compares nylon-1313 performance with published data on nylon-66 (Sweeny and Zimmerman, 1969), the melt viscosity of nylon-1313 is less sensitive to molecular weight effects than is that of nylon-66. In general, the flow properties of molten nylon-1313 are like those of nylons-11 and -610 when preparations having essentially the same viscosity, and nearly the same molecular weight, are compared (Figure 4). Accordingly, we expect that nylon-1313 can be adapted to a wide range of plastic-forming processes without difficulty.

For nylon-1313 at 191 °C and under a shear stress of 6.1×10^5 dyn/cm², the melt viscosity (η) in poise is given by:

$$\log \eta = 1.32 \log \bar{M}_w - 2.98$$

In our studies, molecular weights of nylon-1313 preparations were conveniently estimated by measuring inherent viscosities in *m*-cresol or hexafluoroisopropyl alcohol, a much better

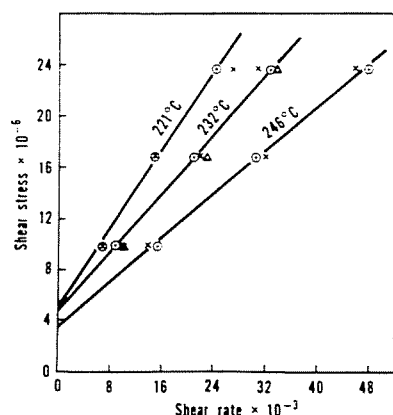


Figure 4. Melt-flow properties of nylon-1313 (O), nylon-11 (X), and nylon-610 (Δ) at different temperatures and shear-stress conditions.

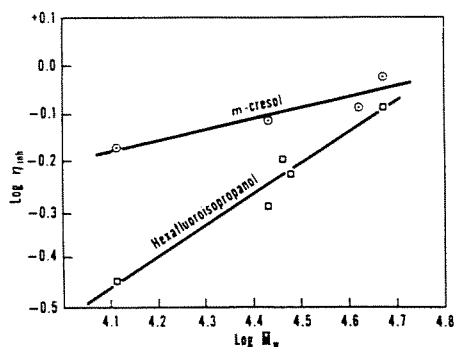


Figure 5. Nylon-1313; logarithm of inherent viscosity (η_{inh}) vs. logarithm of molecular weight (M_w) in 0.5% *m*-cresol and hexafluoroisopropanol.

solvent, and then applying relationships established earlier with preparations whose molecular weights had been determined by light scattering (Figure 5).

Nylon-1313 with an inherent viscosity in the range, 0.7–0.8 dl/g, generally has satisfactory physical properties and reasonable processability. The inherent viscosity should be at least 0.6 dl/g. At this value, the tensile strength of nylon-1313 is close to its maximum. Lower values are accompanied by objectionable losses in tensile strength and, as the viscosity increases above this level, there is only a marginal increase in toughness but a marked loss in ease of processing.

The nylon-1313 prepared in these studies was yellow-colored, although not so much so that there was any difficulty pigmenting the polymer. Neither has there been any noticeable change in unpigmented polymer color with age. To identify the source of this color, each 13-carbon monomer was combined with a nylon-66 monomer, hexamethylenediamine or adipic acid; each resulting polymer was examined visually and a standardized portion was dissolved in hexafluoroisopropanol and characterized in a Cary Model 17 spectrophotometer. The nylon-136 was as good as a top-quality nylon-66, essentially colorless. The nylon-613 was faintly yellow. This relationship was equally apparent by a spectrophotometric measurement used to estimate the color of our polymer preparations.

Polymer	(% T_{650} - % T_{460})/% T_{530}	% T_{530}
Nylon-136	0.05	97
Nylon-613	0.07	92
Nylon-1313	0.08	90

Brassylic acid thus seemed the principal source of color. Our crude acid discolored rapidly when melted in air, and we also

Table III. Process Conditions for Multifilament Melt Spinning

	Nylon-1313	
	Sample A	Sample B
Inherent viscosity, dl/g	1.09	0.60
Extruder feed-zone temp., °C	279	183
Extruder melting-zone temp., °C	290	200
Spinning-block temp., °C	271	202
Melt temp. in block, °C	279	207
Extrusion rate, cm ³ /min	3.7	3.7
Back pressure, psig	2575	525
Yarn take-up rate, m/min	93	128
Yarn denier	1360	1038

Table IV. Orienting Conditions and Yarn Properties

	Nylon-1313	
	Sample A	Sample B
Orienting Conditions		
Platen temperature, °C	160	150
Delivery speed, m/min	28	59–120
Draw ratio	3	5
Yarn Properties		
Denier	453	182
Tenacity, g/d	4.1	6.3
Elongation at break, %	44	15
Initial modulus, g/d	22	40
Tensile factor	22	24

observed a slight color change in the crystalline acid during unprotected storage, which could account for the nylon-1313 being darker than predicted from tests of the monomers.

Processing

Pellets of nylon-1313 suitable for use in most plastic-forming equipment were fabricated directly from the freshly polymerized resin. When white pigmented resin was desired, 1% titanium dioxide (Titanox ACGM 66C18 #3A, Titanium Pigments Div., NL Industries, Inc., South Amboy, N.J.) was added during polymerization, and the resin was pelletized in the same manner. Color was also incorporated into molded items by coating the resin pellets with 0.1% by weight of "Watchung" Red B or Monastral Blue RI (E. I. duPont de Nemours and Co., Inc., Wilmington, Del.) in a Henschel mixer prior to drying and processing.

A variety of items were fabricated without difficulty from the dried nylon-1313 pellets. Data collected during melt spinning of multifilament (Table III) illustrate how processing conditions might be affected by polymer molecular weight or inherent viscosity. Yarns were spun from two lots of nylon-1313 on a 0.75-in. Killian extruder with a 20/1 *L/D* ratio. The polymer was melted in the extruder and transported to the spinning head, an electrically heated block that contained a metering pump, a 1-in. layer of 20–30 mesh Ottawa sand, two screens (250 mesh and 40 mesh), a perforated plate, and the spinneret head, which had 68 orifices of 12 mil diameter with an *L/D* ratio of 2/1. Resin that had an inherent viscosity of 0.6 dl/g extruded quite easily under reasonable temperature and pressure; an inherent viscosity of 1.09 (an 82% increase) required an average 42% higher temperature and a fivefold increase in pressure to achieve the same flow rate. Extruded yarn was air cooled in a 5-ft quench chamber and a 7.5-ft spinway stack and then was passed through conditioning rolls to a Leesona model 959 draw winder. Orientation with the draw winder allowed variation in yarn properties as shown in Table IV. In general, we encountered no difficulties in melt spinning yarn or extruding monofilament from nylon-1313.



Figure 6. Items fabricated from nylon-1313.

Table V. Estimated Fixed Capital Investment for a Plant Producing Nylon-1313 from crude (95%) Brassylic Acid^a

Equipment, delivered	Cost, \$
Equipment for brassylic acid purification	300 000
Units for nitrile production	324 000
Units for diamine production	250 000
Units for salt preparation and polymerization	126 000
Dowtherm units	115 000
Miscellaneous equipment (pumps, tanks, conveyors, etc.)	250 000
Estimated delivered equipment cost	1 365 000
Estimated erected equipment cost	4 800 000
Land and building	700 000
Estimated fixed capital investment	5 500 000

^a Plant capacity: 10 million lb annually, operating 300 day/year, 24 h/day.

Neither was it difficult to extrude film or to injection mold the resin.

Films 5 in. wide and 0.005 in. thick were prepared with a Killian extruder fitted with a 6-in. wide flex-lip die. Resin having an inherent viscosity of 1.09 dl/g required temperatures in the 240–300 °C range at a polymer feed rate of 4 lb/h. Threefold biaxial orientation of the film produced optically clear sheeting with high burst strength. This strength combined with low extractability, high moisture resistance, and heat sealability all should make nylon-1313 film an excellent wrapping material.

The polymer also performed well in trials using a Frohring Model 45 Mini Jector with a cylinder temperature of 245 °C, 5000 psi ram pressure, a 10-s dwell time, and with nylon-1313 that had an inherent viscosity of 0.74 dl/g. Figure 6 shows sample medallions molded from nylon-1313 and other items machined from molded resin. Several of these items were also molded at 275 °C from nylon-1313 having an inherent viscosity of 1.2 dl/g, but cylinder temperatures, which could be varied ± 10 °C, were not critical, and separate heat for the injector nozzle was unnecessary. Both polymer preparations exhibited excellent mold flow, minimal shrinkage, good dimensional stability, and low moisture regain, all of which suit nylon-1313 for a variety of industrial applications.

Estimated Production Costs

An order of magnitude cost estimate for the production of nylon-1313 from crude brassylic acid (95% purity) shows that this material in the form of the molten polymer can be produced for about \$1.00/lb. The estimate is based on a hypothetical plant with an annual capacity of 10 million lb of polymer when operations are conducted 300 days/year, 24 h/day. Predicted yield in the plant is about 83 lb of molten

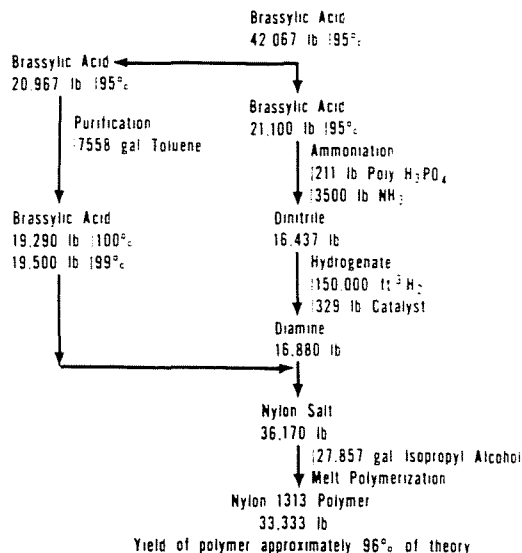


Figure 7. Flowsheet for daily production of nylon-1313.

Table VI. Estimated Cost to Make Nylon-1313 from Crude (95%) Brassylic Acid^a

Cost item	Cents/lb of nylon
Raw materials	
Brassylic acid, 95% @ 54¢/lb	68.1
Other (toluene, ammonia, hydrogen, etc.)	5.9
Total materials costs	74.0
Other direct and indirect costs	
Utilities	1.8
Labor and supervision	7.0
Maintenance and supplies	3.0
Fixed charges	6.7
Charge on working capital	1.7
General plant overhead	5.0
Total direct/indirect costs	25.2
Total cost to make nylon-1313	99.2

^a Plant capacity: 10 million lb annually, operating 300 day/year, 24 h/day.

polymer per 100 lb of crude brassylic acid or the equivalent of about 96% of theory. Estimated fixed capital investment for a battery limits installation is \$5.5 million (Table V). Methods for preparing the estimate followed generally accepted cost estimating procedures and have been described earlier (Sohns, 1971). Costs are reported on a mid-1975 basis.

It is assumed that production of nylon-1313 will be in conjunction with a plant converting crambe oil to brassylic acid and that crude brassylic acid is made available for \$0.54/lb (Carlson et al., 1977). Plant operations in the hypothetical plant in general follow the procedures described previously in this paper. A flowsheet for the daily production of nylon-1313 is shown in Figure 7.

A breakdown of the estimated cost to make is shown in Table VI and includes the following items: raw materials, utilities, labor and supervision, maintenance and supplies, fixed charges, charge on working capital, and general plant overhead.

Cost for brassylic acid when available to the process at \$0.54/lb represents approximately 68% of the cost for producing nylon-1313. The cost of brassylic acid is determined to a large extent by the price of crambe oil. Having brassylic acid available at \$0.54/lb assumes that the delivered price for crambe oil is \$0.30/lb and that appropriate credit is received for byproducts.

The process as described with some probable refinements should be readily adaptable to a commercial operation. Production of dinitriles and diamines from oils other than crambe is already conducted on a plant scale. Other operations in the process are not expected to introduce any serious operating problems.

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