by the demethoxylation with ammonium bromide.[12]

General Procedure for the Oxidative Cleavage of Enol Ethers with RuO_4. To a suspension of α-D-glucal triacetate **7** (270 mg, 1 mmol) dissolved in CCl_4 (10 mL) and $NaIO_4$ (857 mg, 4 mmol) dissolved in water (10 mL) was added $RuO_2 \cdot 2H_2O$ (4 mg, 0.024 mmol). After vigorous stirring for about 10 h, the reaction was quenched with isopropyl alcohol. The organic layer was separated and the aqueous layer was extracted several times with AcOEt. The combined extracts were dried (Na_2SO_4) and the concentrated residue was purified by column chromatography (SiO_2, hexane–AcOEt, 1/1) to give 299 mg (97%) of **8**: mp 128–129 °C: $[\alpha]_D^{17}$ +29.18° (*c* 2.45); IR (Nujol) 3280–2600 (COOH), 1768, 1755, 1748, 1730, 1700 (ester C=O), 1380, 1260, 1240, 1202, 1145, 1110, 1062, 1040, 950, 928, 845, 760 cm^{-1}; ^1H NMR ($CDCl_3$) δ 2.09, 2.14, 2.19 (s, 9, CH_3C=O), 4.27 (m, 2, CH_2O), 5.27–5.83 (m, 3, CHO), 8.05 (s, 1, OCHO), 8.36 (br s, 1, COOH). Anal. Calcd for $C_{12}H_{16}O_{10}$: C, 45.01; H, 5.04. Found: C, 44.96; H, 5.09.

Similar treatment of **7** with a mixture of $RuO_2 \cdot 2H_2O$ (4 mg, 0.024 mmol) and $NaIO_4$ (1286 mg, 6 mmol) for 40 h provided keto acid **9** (276 mg, 96%) which was characterized after esterification with CH_2N_2: (Me ester of **9**) mp 56–57 °C: $[\alpha]_D^{17}$ −10.31° (*c* 6.00); IR (neat) 1750 (ester C=O), 1375, 1220, 1070 cm^{-1}; ^1H NMR ($CDCl_3$) δ 2.15 (s, 6, CH_3COO), 2.17 (s, 3, CH_3COO), 3.75 (s, 3, OCH_3), 4.85 (d, *J* = 2 Hz, 2, CH_2O), 5.59 (d, *J* = 2.5 Hz, 1, OCHCO), 5.73 (d, *J* = 2.5 Hz, 1, OCHCOO). Anal. Calcd for $C_{12}H_{16}O_9$: C, 47.38; H, 5.30. Found: C, 47.31; H, 5.33.

Conversion of 8 into 9. A mixture of **8** (160 mg, 0.5 mmol), $RuO_2 \cdot 2H_2O$ (3 mg, 0.02 mmol), and $NaIO_4$ (642 mg, 3.0 mmol) was added to CCl_4 (10 mL) and H_2O (10 mL). The resulting suspension was vigorously stirred for 45 h and the products were taken up in AcOEt. Usual workup followed by column chromatography (SiO_2, hexane–AcOEt, 1/1) gave 130 mg (90%) of **9**.

Results of the oxidation of enolic olefins with RuO_4 are given in Table I and physical properties along with spectral data of products are as follows.

6: bp 139–141 °C (1 mm); IR (neat) 3600–2400 (COOH), 1720 (C=O), 1410, 1180, 1085 cm^{-1}; ^1H NMR ($CDCl_3$) δ 0.90 (m, 3, CH_3), 1.10–2.75 (m, 10, CH_2), 5.05 (m, 1, CHO), 8.09 (s, 1, OCHO). Anal. Calcd for $C_9H_{16}O_4$: C, 57.43; H, 8.57. Found: C, 57.52; H, 8.51.

11: bp 128–130 °C (2 mm); IR (neat) 1740 (C=O), 1715 (C=O), 1405, 1355, 1210, 1170, 965 cm^{-1}; ^1H NMR ($CDCl_3$) δ 1.00–1.75 (m, 7, CH_2, CH), 1.90–2.75 (m, 4, CH_2, CH), 2.13 (s, 3, CH_3C=O), 4.85 (d, *J* = 6 Hz, 1, CHO), 7.95 (s, 1, OCHO). Anal. Calcd for $C_{11}H_{16}O_3$: C, 67.32; H, 8.22. Found: C, 67.30; H, 8.27.

13: bp 144–146 °C (2 mm); IR (neat) 3400–2300 (COOH), 2640 (CHO), 1720 (C=O), 1695 (COOH), 1460, 1405, 1300, 1245, 1170, 900, 680 cm^{-1}; ^1H NMR ($CDCl_3$) δ 1.00–2.50 (m, 11, CH_2, CH), 4.90 (m, CHO), 7.98 (s, 1, OCHO), 10.48 (s, 1, COOH). Anal. Calcd for $C_{10}H_{14}O_4$: C, 60.59; H, 7.12. Found: C, 60.62; H, 7.19.

16: mp 54–55 °C; IR (Nujol) 1715 (C=O), 1697 (C=O), 1455, 1378, 1255, 1060, 1010, 1000 cm^{-1}; ^1H NMR ($CDCl_3$) δ 0.96 (d, *J* = 7 Hz, 6, CH_3), 1.10–2.00 (m, 4, CH_2, CH), 2.00–2.80 (m, 6, CH_2), 3.47–4.55 (m, 2, CH_2O). Anal. Calcd for $C_{11}H_{18}O_3$: C, 66.64; H, 9.15. Found: C, 66.59; H, 9.16.

18: bp 146–150 °C (2 mm); IR (neat) 3640–2400 (COOH), 1732 (C=O), 1705 (C=O), 1690 (C=O), 1320, 1270, 1230, 1185, 1012, 772 cm^{-1}; ^1H NMR ($CDCl_3$) δ 1.13–1.50 (m, 6, CH_3), 1.80–2.50 (m, 4, CH_2), 4.07–4.74 (m, 3, CH_2, CH), 9.21 (br s, 2, CHO, COOH). Anal. Calcd for $C_9H_{15}O_5$: C, 49.76; H, 6.96. Found: C, 49.76; H, 6.86.

20: bp 139–142 °C (2 mm); IR (neat) 1740 (C=O), 1712 (C=O), 1695 (C=O), 1385, 1372, 1247, 1178, 1170, 1045, 920, 732 cm^{-1}; ^1H NMR ($CDCl_3$) δ 1.02 (d, *J* = 6 Hz, 3, CH_3), 1.55–2.84 (m, 11, CH_2, CH), 2.38 (s, 3, CH_3C=O), 3.70–4.03 (m, 2, CH_2NC=O). Anal. Calcd for $C_{12}H_{19}NO_3$: C, 63.98; H, 8.50. Found: C, 63.90; H, 8.52.

Cleavage of Enol Acetate 21 with a Stoichiometric Amount of RuO_4. To a cooled (0 °C) solution of menthone enol acetate **21** (196.3 mg, 1 mmol) in CCl_4 (10 mL) was added dropwise a yellow solution of RuO_4 generated from $RuO_2 \cdot 2H_2O$ (338 mg, 2 mmol) and $NaIO_4$ (856 mg, 4 mmol) in CCl_4 (50 mL). The mixture was stirred for 1 h at 0–5 °C and the reaction was quenched with isopropyl alcohol (1 mL). The mixture was freed from RuO_2 by filtration under reduced pressure and the filtrate was concentrated to give a mixture of **22**[13] (29 mg, 16%) and **23** (160 mg, 80%) after purification on column chromatography (SiO_2, hexane–AcOEt, 4/1).

Treatment of 22 with RuO_4 Regenerated from $RuO_2 \cdot 2H_2O$ (Catalytic) and $NaIO_4$ (Stoichiometric). Hydroxy ketone **22** (100 mg, 0.66 mmol), $NaIO_4$ (561 mg, 2.62 mmol), and $RuO_2 \cdot 2H_2O$ (3 mg, 0.02 mmol) were placed in CCl_4 (10 mL) and H_2O (10 mL). The mixture was stirred vigorously for 1 h at 0–5 °C and quenched with 2-propanol (0.5 mL). Extractive workup followed by column chromatography (SiO_2, hexane–AcOEt, 2/1) gave 89 mg (89%) of **22** and 10 mg (9%) of **23**.[13]

Lactonization of Formyloxy Carboxylic Acid. A solution of **13** (66 mg, 0.33 mmol) and a catalytic amount of *p*-TsOH in benzene (1 mL) was heated to reflux for 30 min. The mixture was washed with aqueous $NaHCO_3$ and dried (Na_2SO_4). Evaporation followed by column chromatography (SiO_2, hexane–AcOEt, 1/1) gave 41.6 mg (82%) of lactone **14**: bp 105–107 °C (1 mm); IR (neat) 1775 (lactone C=O), 1460, 1415, 1365, 1310, 1215, 1175, 1115, 1045, 1030, 890, 875, 785, 685 cm^{-1}; ^1H NMR ($CDCl_3$) δ 1.00–2.90 (m, 11, CH_2, CH), 4.48 (m, 1, OCH). Anal. Calcd for $C_9H_{12}O_2$: C, 71.03; H, 7.95. Found: C, 70.96; H, 7.97.

Registry No. **1**, 2270-61-3; **2**, 94924-63-7; **3**, 64833-76-7; **4**, 1679-47-6; **5**, 60443-97-2; **6**, 94924-64-8; **7**, 2873-29-2; **8**, 94924-61-5; **9**, 98919-96-1; **9** (methyl ester), 98839-00-0; **10**, 98838-97-2; **11**, 98838-98-3; **12**, 69486-16-4; **13**, 98838-99-4; **14**, 5963-22-4; **15**, 94924-57-9; **16**, 94924-51-3; **17**, 94924-59-1; **18**, 94924-54-6; **19**, 94924-60-4; **20**, 94924-55-7; **21**, 20144-45-0; **22**, 74219-28-6; **23**, 589-60-6; RuO_4, 20427-56-9.

An *N*-Carboxyanhydride (NCA) Route to Aspartame†

Jacob S. Tou* and Billy D. Vineyard

Monsanto Company, Nutrition Chemicals Division, St. Louis, Missouri 63167

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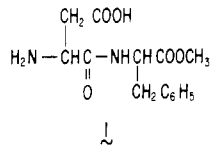
α-L-Aspartyl-L-phenylalanine methyl ester **1** (aspartame) is a nutritive sweetener approximately 200 times as sweet as sucrose.[1] Since the discovery in 1969, much effort has been directed toward an efficient synthesis.[2]



The selective formation of a peptide bond in the α-position of the L-aspartic acid moiety poses a major challenge to the synthesis of aspartame. It has been shown that the ring-opening reaction of N-substituted L-aspartic anhydride **2** with L-phenylalanine or its methyl ester **3** gives a mixture of α- and β-adducts **4** and **5**, with a predominance of the α-isomer[2] (Scheme I). Therefore, a separation/recovery step is required.[2] On the other hand, several regioselective routes to the α-dipeptide have also been reported by either enzymatic[3a,b] or chemical methods.[3c] For example, the approach by Vinick[3c] et al. involves the coupling of L-phenylalanine methyl ester **3** and L-aspartic acid *N*-thiocarboxyanhydride (NTA) **6**, which was prepared from L-aspartic acid and methyl ethyl xanthate followed by PBr_3 cyclization.

We now wish to report our discovery of a simple and regioselective synthesis of aspartame from β-methyl-L-aspartate *N*-carboxyanhydride **7** (NCA) (Scheme II). This

† After this work had been completed in our laboratory, a similar but independent result was recently reported in a patent application (*Chem. Abstr.* **1985**, *102*, 96095*h*).

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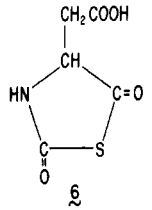

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route involves a regioselective coupling of NCA 7 with L-phenylalanine and a selective precipitation of α-L-aspartyl-L-phenylalanine methyl ester hydrochloride 9.

Results and Discussion

β-Methyl-L-aspartate *N*-carboxyanhydride 7 was prepared in two steps. Esterification of L-aspartic acid in methanol–HCl is highly selective[4] with a β/α ratio greater than 8. The resulting β-methyl-L-aspartate hydrochloride was reacted with phosgene[4] to form the crystalline NCA 7. Unlike the unstable *N*-carboxyanhydride of aspartic acid,[5a] the NCA of its β-methyl ester 7 used here is fairly stable.

The regioselective reaction of phenylalanine and 7 was conducted in an aqueous medium with pH controlled[5] at 10.0–10.2. The resulting coupling adduct 8, the aspartyl methyl ester of α-L-aspartyl-L-phenylalanine, was subjected to a hydrolysis–esterification reaction without isolation. α-L-Aspartyl-L-phenylalanine hydrochloride 10 was first formed, which then entered an esterification reaction (Scheme III). A series of equilibria was established with all the possible esterification products present.[2a] The desired α-L-aspartyl-L-phenylalanine methyl ester hydrochloride 9 was essentially insoluble in the reaction medium and precipitated as formed. The diacid and the other esters 8 and 11 are very soluble. Thus, the reaction was driven to 9. It is noteworthy that the rate of formation of 9 depends upon the degree of hydrolysis of 8 to diacid before entering the esterification medium.[6] This hydrolysis–esterification procedure transposes the β-ester function in 8 to the phenylalanine carboxyl as in 9. The conversion of 9 to 1 by a neutralization procedure completed this NCA route to aspartame.

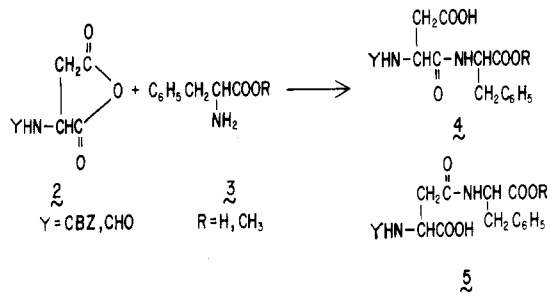
Our synthesis of aspartame uses inexpensive materials and avoids the repugnant sulfur reagent.[3c] This novel sweetener is prepared in good yield with complete regiochemical control.

Experimental Section

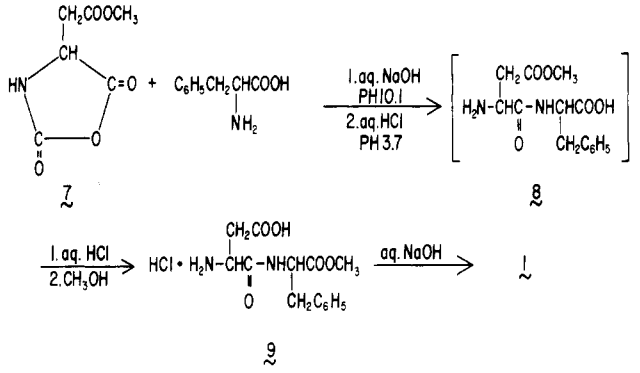
General. Melting points were determined on a Thomas-Hoover capillary melting point apparatus and were uncorrected. ^{1}H NMR spectra were recorded on a Varian T-60 spectrometer. The specific rotations [α] were determined on a Perkin-Elmer Model 141 polarimeter. Reaction progress was monitored with a liquid chromatograph (Water Associates) equipped with a UV detector (210 nm). Microanalyses were performed by the Physical Science Center of Monsanto Co.

Starting Materials. β-Methyl-L-aspartate hydrochloride was prepared by the method of Coleman[4a] from L-aspartic acid and anhydrous methanolic hydrogen chloride. After removal of most of the methanol, pure β-isomer was selectively precipitated by addition of ethyl acetate to give 78% yield: mp 194–196 °C; $[\alpha]^{20}_D$ 12.9° (*c* 1.0, 1:3 ethanol–water). β-Methyl-L-aspartate *N*-carboxyanhydride 7 was prepared by the following modified Coleman's procedure.[4a] Gaseous phosgene (95 g, 0.96 mol) was bubbled into a slurry of β-methyl-L-aspartate hydrochloride (80 g, 0.44 mol) in 800 mL of THF. The mixture was then heated at 60 °C for 2 h. A rapid stream of nitrogen was passed through the solution to remove excess phosgene. Solvent was stripped, and the colorless residue was placed in an ice bath. To this was added 40 mL of ethyl acetate followed by 90 mL of petroleum ether. The product precipitated to give 80–85% (64 g) isolated yield: mp 59–61 °C (lit.[4b] mp 80 °C dec); $[\alpha]^{22}_D$ −71.7° (*c* 3.0, chloroform) (lit.[4b] $[\alpha]^{25}_D$ −72.8° (*c* 3.0, chloroform)). Anal. Calcd

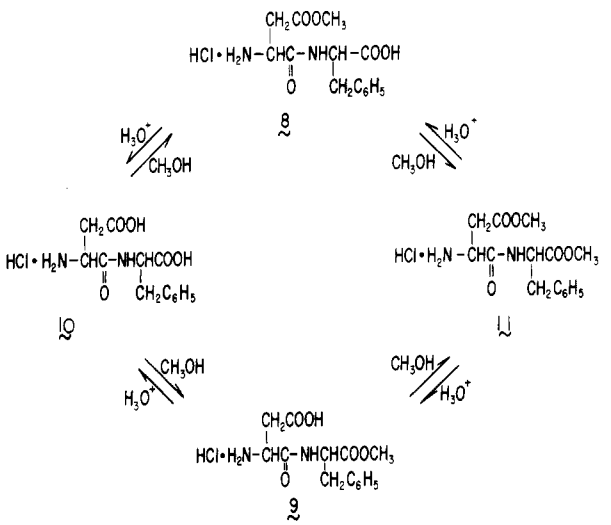
Scheme I



Scheme II



Scheme III



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(6) It was demonstrated that diacid 10 was a better intermediate than ester 8, since conversion to and precipitation of 9 was much faster with 10.

for $C_6H_7O_4N$: C, 41.63; H, 4.08; N, 8.09. Found: C, 41.71; H, 3.97; N, 7.82.

Preparation of α-L-Aspartyl-L-phenylalanine Methyl Ester Hydrochloride 9. L-Phenylalanine (7.4 g, 0.045 mol) was stirred in 90 mL of water. The pH of this solution was adjusted to 10.2 (0–2 °C) with 50% NaOH. Then a solution of pure NCA 7 (8.3 g, 0.048 mol) in 8 mL of THF was added in 15 min with vigorous stirring. The pH was maintained at 10.0–10.2 by the addition of 7 N NaOH solution. The reaction mixture was then stirred at 0–2 °C for 2 h (pH 10.0–10.2). One equivalent of 37% hydrochloric acid (9.7 g) was added at the end of the hold period. Liquid chromatography indicated an 80–82% yield of aspartyl ester 8 based on L-phenylalanine. This clear solution was extracted twice with 50-mL portions of ethyl acetate. To the aqueous solution, 4.2 g of 37% HCl (0.043 mol) was added. The solution was concentrated in vacuo to a total weight of 31.1 g. Another 8.4 g (0.085 mol) of 37% HCl was added, and the reaction slurry was held at 40 °C for 6 h to convert 8 to α-L-aspartyl-L-phenylalanine hydrochloride **10**. Solid (NaCl) was collected and washed with 2.6 g (0.026 mol) of 37% HCl and 7.5 g of methanol. Seed crystals were added to the combined filtrate and washings. The resulting solution was stirred at ambient temperature for 68 h. The thick slurry was cooled to 0–2 °C, and the solid was collected by filtration and washed with 7 mL of cold water. The dry weight of **9** was 9 g (55% yield[7] based on L-phenylalanine). This material was then neutralized with sodium hydroxide to give aspartame 1,[2a] $[\alpha]^{20}_D$ 30.3° (*c* 1.0, HOAc), authentic sample, $[\alpha]^{20}_D$ 30.1° (*c* 1.0, HOAc).

Registry No. 1, 22839-47-0; 7, 21933-62-0; 8, 22839-82-3; 9, 5910-52-1; L-aspartic acid, 56-84-8; β-methyl-L-aspartate hydrochloride, 16856-13-6; phosgene, 75-44-5; L-phenylalanine, 63-91-2.

(7) The HPLC analysis of the mother liquor indicated the presence of a 2:2:1 ratio of **8**, **9**, and diacid **10**. A small amount of phenylalanine and diester **11** were also observed. It is likely that recycling the mother liquor would raise the overall yield of **9** above the current 55%.

N-Methyloxazolinium Salts: Diastereomer Ratios by ^{1}H NMR

Mark. J. Kurth,* Edward G. Brown, and Owen H. W. Decker

Department of Chemistry, University of California, Davis, Davis, California 95616

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The oxazoline heterocycle is very useful in synthesis, particularly as a transient chiron[1,2] in enantioselective methodology,[3,4] The expanding structural complexity of chiral products made available by the numerous oxazoline-based synthetic methods complicates the analytical problem of assessing the enantioselectivity obtained in these various transformations. As is inherent to all enantioselective methods employing a transient chiron, the chiral products obtained in each oxazoline-based variant are diastereomeric in nature. Thus, prior to liberation of the transient chiron, they accommodate a variety of methods for assessing the degree of asymmetric induction (i.e., diastereoselectivity).

The two analytical methods most successfully used in oxazoline-based studies are high-pressure liquid chromatography (HPLC) and ^{1}H NMR. Of the two, HPLC has generally been the analytical tool of choice because it is versatile, sensitive, and reliable. Unfortunately, its application to a broad range of substrates can be complicated, and often requires experimentation with a large number of column and solvent variations. This is particularly true for nonrigid oxazolines in which the chiral centers are separated by several atoms.

Given the ease of sample preparation and the availability of high-field instrumentation, direct ^{1}H NMR analysis of oxazoline diastereomers would appear to be an attractive alternative. We find, however, that only rarely are baseline-resolved diastereomeric resonances observed (vide infra). Indeed most ^{1}H NMR derived ratios have been determined by chiral lanthanide-induced shift (LIS) studies on the enantiomers after the transient chiron has been removed.[5] LIS studies, although versatile, are limited by the vicissitudes of fortune and the paramagnetic line broadening inherent in the method.[6] This loss of resolution due to line broadening is particularly troublesome in the assessment of product ratios from highly enantioselective methods.

Our aza-Claisen work,[4] which generates oxazolines with chiral centers separated by two or three atoms required a rapid, general method for diastereomer ratio determination. While HPLC resolves a number of these diastereomers, we have found that baseline resolution via HPLC is capricious, particularly when the chiral centers are separated by three atoms. Faced with the time-consuming prospect of generating a unique HPLC protocol for each new substate, we set out to develop a rapid and reliable ^{1}H NMR method of assessing oxazoline-based diastereomer ratios.

In the course of developing our aza-Claisen procedure, we prepared a series of *N*-allyloxazolinium salts. In each case, a number of significant chemical shift changes were noted in the ^{1}H NMR spectrum of the salt relative to the unalkylated oxazoline. While downfield shifts were anticipated for protons proximal to the delocalized charge,[7] a number of more remote protons displayed unexpected shifts. These "aberrant" chemical shift changes may be the result of restricted rotation about the C(2)–C(α) bond caused by conformational restraints imparted by the N-allyl substituent.[8] Extending this observation to diastereomeric oxazolines, we reasoned that *N*-alkylation would amplify the conformational bias of each diastereomer and might, therefore, afford baseline resolution of peaks in the ^{1}H NMR.

This proved to be the case. For example, the 360 MHz ^{1}H NMR spectrum of a diastereomeric mixture of N-allyloxazolinium salts **1** displayed a number of base-line-resolved resonances (Table I). In contrast, a diastereomeric mixture of (4*S*,1′*R*)- and (4*S*,1′*S*)-4,5-dihydro-4-(1-methylethyl)-2-(1-phenylethyl)oxazole gave no base-line-resolved ^{1}H NMR resonances.

Although *N*-allyl salts could be used to determine diastereomer ratios in several cases, generally they were not useful due to the complexity of their ^{1}H NMR spectra. We therefore turned to the *N*-methyl derivatives and were delighted to find that these salts not only afforded much simpler NMR spectra, but also were formed faster, under

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