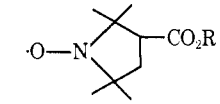
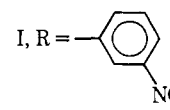
esters *via* the intermediate formation of inclusion complexes.

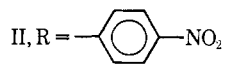
Rate measurements on the hydrolyses of phenyl esters have provided evidence[4] that catalysis of these reactions by the cycloamyloses (cyclodextrins) occurs by the kinetic scheme illustrated by eq 1 which is similar to that demonstrated to hold for catalysis by α-chymotrypsin.[1,5]

$$S + CA \underset{K_d}{\rightleftharpoons} S\cdot CA \xrightarrow{k_2} S'CA \xrightarrow{k_3} P_2 + CA \quad (1)$$
$$+ P_1$$

The substrate employed in the present work was the ester I. A similar compound II reacts with serine-195 at the active site of α-chymotrypsin to give a spin-labeled acyl enzyme species.[6]







III, R = cycloheptaamylose

IV, R = H

From visible absorption measurements on the rate of production of the *m*-nitrophenolate ion from I at pH 9.7 (carbonate buffer) and 25.0° in the presence of varying amounts of excess cycloheptaamylose we calculated a value of $K_d = 7.5 \pm 0.6 \times 10^{-4}$ *M* and $k_2 = 6.9 \times 10^{-3}$ sec^{-1}.

From the work of Bender, *et al.*,[4] the value of K_d would not be expected to be significantly different in acidic solution. When a solution of I (10^{-4} *M*) and cycloheptaamylose (4×10^{-3} *M*) in phosphate buffer at pH 5.75 was examined using a Varian E-3 spectrometer the esr signal illustrated in Figure 1a was observed. For comparison the esr spectrum of I in the absence of the cycloamylose is shown in Figure 1b. The rotational correlation time τ for I was calculated[7] to be 0.35×10^{-10} sec. The spectrum of Figure 1a is attributed to substrate–cycloheptaamylose "Michaelis" complex (S·CA) with $\tau = 3.34 \times 10^{-10}$ sec. *m*-Nitrophenolate ion did not form in an appreciable quantity during the course of the esr measurement. By varying the concentration of excess cycloheptaamylose the dissociation constant K_d was estimated as $6 \pm 2 \times 10^{-4}$ *M* at pH 5.75, in good agreement with the value obtained from the kinetic studies at pH 9.7.

The acyl cycloheptaamylose III was prepared, using the procedure described by Bender,[4] from cycloheptaamylose and I at pH 9.6. Gel filtration chromatography (G-10 Sephadex, pH 5.75 phosphate buffer) was employed to separate the required intermediate from the unreacted ester and the products of hydrolysis.

The value of τ calculated from the esr spectrum observed for a solution of the acyl cycloheptaamylose III (Figure 1c) at pH 5.75 was 5.04×10^{-10} sec. A comparison of this rotational correlation time with the one measured for the "Michaelis" complex indicates that the spin label is somewhat more immobilized in III than in the noncovalent complex.

On raising the pH to 9.6 the rate of deacylation of III was studied by following the increase in height of the high-field hyperfine component of the spectrum, as the more rapidly tumbling acid IV is formed. The rate constant for the deacylation step was calculated as $k_3 = 3.2 \times 10^{-5}$ sec^{-1}.

In summary, using the ester substrate I, we have observed directly the "Michaelis" complex S·CA and the acyl cycloheptaamylose complex S'CA by esr spectroscopy. The rotational correlation time τ for the "Michaelis" complex was found to be intermediate between those found for the uncomplexed substrate I and the acyl cycloheptaamylose III, although it was closer to the one for the latter species. Specificity in the action of proteolytic enzymes has been interpreted by Bender, *et al.*,[8] in terms of an interaction of the R' group of the acyl function (R'C=O) with the enzyme surface, rigidifying the whole set of bonds involved in the reaction so that the acyl group occupies the correct position for reaction even in the ground state. In agreement with this picture it is quite clear that the R' group of the acyl function of the ester I, the part of the substrate containing the nitroxide moiety, is significantly immobilized in the "Michaelis" complex with the model enzyme cycloheptaamylose. The results of esr measurements on Michaelis complexes of substrates with the cycloamyloses and with proteolytic enzymes will provide a critical test for the proposed theory of enzyme specificity.

Acknowledgment. The support of the Petroleum Research Fund of the American Chemical Society is gratefully acknowledged. We also wish to thank Professor F. J. Kezdy for a helpful discussion.

(5) In eq 1, S represents the ester, CA the cycloamylose, S·CA the inclusion or "Michaelis" complex, S'CA the acylcycloamylose, P_1 the product alcohol, and P_2 the product acid.

(6) L. J. Berliner and H. M. McConnell, *Proc. Natl. Acad. Sci. U. S.*, **55**, 708 (1966).

(7) D. Kivelson, *J. Chem. Phys.*, **27**, 1087 (1957); J. H. Freed and G. K. Fraenkel, *ibid.*, **39**, 326 (1963).

(8) M. L. Bender, F. J. Kezdy, and C. R. Gunter, *J. Amer. Chem. Soc.*, **86**, 3714 (1964).

(9) Fellow of the Alfred P. Sloan Foundation.

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Received May 7, 1970

Methoxy Groups As Probes for Delocalized Cations. Substituent Effects on 2-Norbornyl Solvolysis Rates

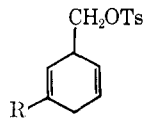
Sir:

The effect on solvolysis rates by substituents remote from the reaction site is a standard way to detect the presence or absence of charge delocalization.[1] As the following examples (I–IV) suggest, methoxy groups may be more sensitive[2] for this purpose than the commonly employed methyl groups.[1,3–8]

(1) A. Streitwieser, "Solvolytic Displacement Reactions," McGraw-Hill, New York, N. Y., 1962; D. Bethell and V. Gold, "Carbonium Ions," Academic Press, New York, N. Y., 1967.

(2) *Cf.* T. G. Traylor and J. C. Ware, *J. Amer. Chem. Soc.*, **89**, 2304 (1967); *Tetrahedron Lett.*, 1295 (1965); R. H. Martin, F. W. Lampe, and R. W. Taft, *J. Amer. Chem. Soc.*, **88**, 1353 (1966).

(3) *E.g.*, S. Winstein, C. R. Lindegren, H. Marshall, and L. L. Ingraham, *ibid.*, **75**, 147 (1953); R. A. Sneen, *ibid.*, **80**, 3982 (1968); P. D. Bartlett and G. D. Sargent, *ibid.*, 87, 1297 (1965); K. L. Servis and J. D. Roberts, *ibid.*, 87, 1331 (1965).

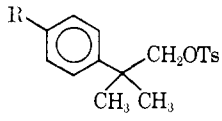


I

R	k_{rel}[4]
H	1
CH_3	22
OCH_3	2400

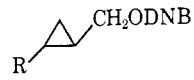
II

R	k_{rel}[5]
H	1
CH_3	7.8
OCH_3	97



III

R	k_{rel}[6]
H	1
CH_3	7.3
OCH_3	89



IV

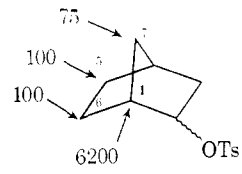
R	k_{rel}[7]
H	1
CH_3	11
OCH_3	791

As a consequence, we hoped that methoxy groups would be effective in detecting small amounts of positive charge, even in systems where methyl substituents failed to give clean-cut results.[8] We have chosen the 2-norbornyl system for a test and have prepared a variety of methoxy-substituted derivatives.

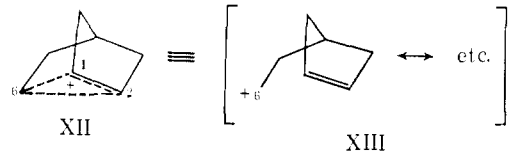
Hydroboration of 5-*exo*-methoxy-2-norbornene[9] (V) gave a mixture of ~40% of 5-*exo*- (VI) and ~60% of 6-*exo*-methoxy-2-*exo*-norbornanol (VII). These isomers could be separated by preparative gas chromatography. Oxymercuration of V gave a different ratio of the same products: ~92% VI and ~8% VII. Unambiguous structural assignments were made on the basis of the nmr spectra of the corresponding dimethyl ethers: 2-*exo*-5-*exo*-dimethoxynorbornane gave but a single bridgehead peak while the 2,6 isomer showed two well-separated peaks. *anti*-7-Hydroxynorbornene[10] was converted to the methyl ether (Williamson synthesis) and hydroborated to VIII. Lithium aluminum hydride reduction of the epoxide prepared from 1-methoxynorbornene[11] gave a mixture of comparable amounts of 1-methoxy-2-*exo*-norbornanol (IX) and 4-methoxy-2-*exo*-norbornanol (X). These isomers, when separated by spinning band column distillation, could be distinguished in the infrared: only IX gave an intramolecular hydrogen bond. Chromic acid oxidation of alcohols VI–IX to the corresponding ketones, followed by $LiAlH_4$ reduction, sufficed to prepare the methoxy-substituted 2-*endo*-norbornanols. Tosylates were prepared by the usual pyridine method.

Acetolysis rate constants and relative rates are summarized in Table I. It is clear that in most positions the methoxy groups, due to their electron-withdrawing inductive effects, produce rate depressions the magnitudes of which are consistent with those observed in other alkyl systems where an electronegative substituent is isolated from the reaction center by one or more carbon atoms and no heteroatom participation occurs.[12-20] Gassman has included the data on 7-*anti*-methoxy-2-*exo*- and 2-*endo*-norbornyl tosylates in his analysis of inductive effects of 7 substituents in the norbornyl system,[17b] where it was shown that the p^* for *exo* solvolysis was somewhat larger than p^*-*endo*. In the present instance 7-*anti*-, 5-*exo*-, and 6-*exo*-methoxy substituents cause similar effects—the rate depressions observed for 2-*exo*-tosylate solvolysis are about three times larger than those for the corresponding 2-*endo*-tosylates.

The positional responses of the *exo*/*endo* rate ratios (depicted in XI) are the most concise way of analyzing the results. Methoxy substitution of the 5-*exo*, 6-*exo*, and 7-*anti* derivatives produces only a slight decrease in the *exo*/*endo* ratio from the parent compound (*exo*/*endo* = 300). These results afford no evidence for significant charge delocalization to the 6 position in the



XI

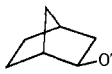
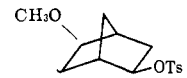
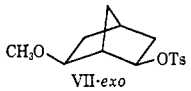
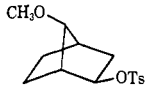
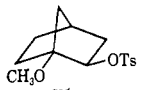
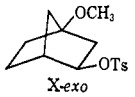
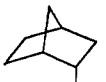
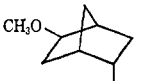
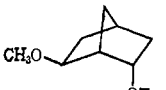
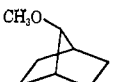
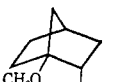


XII XIII

transition state for 2-*exo*-norbornyl solvolysis.[8] This implies that structure XIII either does not contribute significantly to the resonance hybrid[8,21] or that 6-methoxy groups are incapable of detecting such contributions.

(4) O. L. Chapman and P. Fitton, *J. Amer. Chem. Soc.*, 85, 41 (1963).
(5) H. Felkin and C. Lion, *Chem. Commun.*, 60 (1968).
(6) R. Heck and S. Winstein, *J. Amer. Chem. Soc.*, 79, 3432 (1957).
(7) P. v. R. Schleyer and G. W. van Dine, *ibid.*, 88, 2321 (1966); H. Alper, unpublished observations.
(8) *Cf.* P. v. R. Schleyer, M. M. Donaldson, and W. E. Watts, *ibid.*, 87, 375 (1965).
(9) S. J. Cristol, W. K. Seifert, D. W. Johnson, and J. B. Jurale, *ibid.*, 84, 3918 (1962).
(10) P. R. Story, *J. Org.Chem.*, 26, 287 (1961).
(11) Prepared from 1-acetoxynorbornene (J. W. Wilt, C. F. Parsons, C. A. Schneider, D. G. Schultenover, S. J. Wagner, and W. J. Wagner, *ibid.*, 33, 694 (1968)) by cleavage of the 1-acetate with $LiAlH_4$ followed by methylation.
(12) D. S. Noyce and H. I. Weingarten, *J. Amer. Chem. Soc.*, 79, 3103 (1957); D. S. Noyce, B. R. Thomas, and B. N. Bastian, *ibid.*, 82, 885 (1960); D. S. Noyce, B. N. Bastian, and R. S. Monson, *Tetrahedron Lett.*, 863, (1962); H. Kwart and T. Takeshita, *J. Amer. Chem. Soc.*, 86, 1161 (1964); E. W. Della and P. R. Jeffries, *Aust. J. Chem.*, 14, 610 (1961).
(13) (a) D. C. Kleinfelter and P. von R. Schleyer, 138th National Meeting of the American Chemical Society, New York, N. Y., Sept 1960, Abstracts, p 43Z; D. C. Kleinfelter, *Diss. Abstr.*, 22, 428 (1961); (b) J. A. Berson in "Molecular Rearrangements," Part I, P. de Mayo, Ed., Interscience, New York, N. Y., 1963, p 182.
(14) W. R. Vaughan, R. Caple, J. Csapilla, and P. Scheiner, *J. Amer. Chem. Soc.*, 87, 2204 (1965).
(15) P. v. R. Schleyer, *ibid.*, 89, 3901 (1967).
(16) J. W. Wilt and W. J. Wagner, *ibid.*, 90, 6135 (1968).
(17) (a) P. G. Gassman and J. M. Hornback, *ibid.*, 91, 4280 (1969); (b) P. G. Gassman, J. L. Marshall, J. G. Macmillan, and J. M. Hornback, *ibid.*, 91, 4282 (1969).
(18) H. Goering and M. J. Degani, *ibid.*, 91, 4506 (1969).
(19) A. J. Fry and W. B. Farnum, *J. Org. Chem.*, 34, 2314 (1969).
(20) R. Muneyuki and T. Yano, *J. Amer. Chem. Soc.*, 92, 746 (1970).
(21) A similar conclusion has been reached on the basis of an analysis of nmr ^1H and ^{13}C chemical shifts: G. Olah and A. M. White, *ibid.*, 91, 6883 (1969). Also see, *e.g.*, R. Howe, E. C. Friedrich, and S. Winstein, *ibid.*, 87, 379 (1965), where the point is made that a bridging carbon (C-6 in norbornyl) should tend toward sp^3 hybridization and should bear little excess positive charge.

Table I. Acetolysis Rates of Methoxy-Substituted Norbornyl *p*-Toluenesulfonates[a]

Tosylate	Temp, °C	k, sec^{-1}	k_{rel}, 25°	$\Delta H^{\pm}$, kcal/mol	$\Delta S^{\pm}$
	25[b]	2.40×10^{-5}	298	22.4	−4.6
 VI-*exo*	25[c]	3.27×10^{-7}	4.0	26.5	−0.8
	50.1	$1.02 \pm 0.08 \times 10^{-5}$			
	74.9	$2.31 \pm 0.12 \times 10^{-4}$			
	84.6	$5.13 \pm 0.25 \times 10^{-4}$			
	100.0	$2.65 \pm 0.15 \times 10^{-3}$			
 VII-*exo*	25[c]	3.3×10^{-6}	40.0	22.2	−9.1
	50.2	$6.1 \pm 0.6 \times 10^{-5}$			
	74.9	$7.1 \pm 0.7 \times 10^{-4}$			
 VIII-*exo*	25[c]	1.95×10^{-6}	24	23.5	−5.7
	50.2	$4.32 \pm 0.51 \times 10^{-5}$			
	74.9	$5.82 \pm 0.56 \times 10^{-4}$			
 IX-*exo*	25[c]	2.98×10^{-5}	366	22.3	−4.5
	25.0[d]	$2.82 \pm 0.16 \times 10^{-5}$			
	50.1[d]	$7.50 \pm 0.25 \times 10^{-4}$			
	75[d]	$7.30 \pm 0.30 \times 10^{-3}$			
 X-*exo*	25[c]	1.23×10^{-6}	15.1	24.2	−4.4
	50.1	$3.18 \pm 0.20 \times 10^{-5}$			
	74.9	$5.01 \pm 0.10 \times 10^{-4}$			
	25[b]	8.14×10^{-8}	1.0	26.5	−2.0
 VI-*endo*	25[c]	3.54×10^{-9}	0.04	28.8	−4.7
	75.1	$3.9 \pm 0.4 \times 10^{-6}$			
	100.8	$6.84 \pm 1.0 \times 10^{-5}$			
 VII-*endo*	25[c]	3.26×10^{-8}	0.40	27.7	0.0
	84.6	$7.86 \pm 0.29 \times 10^{-5}$			
	100.8	$4.25 \pm 0.11 \times 10^{-4}$			
 VIII-*endo*	25[c]	3.08×10^{-8}	0.38	27.5	−0.8
	84.6	$6.96 \pm 0.27 \times 10^{-5}$			
	100.8	$3.71 \pm 0.08 \times 10^{-4}$			
 IX-*endo*	25[c]	4.82×10^{-9}	0.06	26.1	−9.0
	100.0[d]	$4.27 \pm 0.01 \times 10^{-5}$			
	124.9[d]	$4.20 \pm 0.50 \times 10^{-4}$			

[a] Rates determined titrimetrically unless otherwise noted. [b] Calculated from data in ref 6. [c] Extrapolated from higher temperatures. [d] Determined conductometrically.

The relatively large increase in the *exo/endo* rate ratio produced by 1-methoxy substitution[22] falls into the pattern observed for other 1 substituents (Table II). Existing data on the solvolysis of such 1-substituted norbornyl derivatives give rather good σ^* plots; the magnitude of $p^*_{CH_2}$-*exo* (~ -5)) is about *twice* as large as $p^*_{CH_2}$-*endo* (~ -2.5). The larger magnitude of $p^*_{CH_2}$-*exo* may be due to a greater degree of charge

(22) Y. Lin and A. Nickon have found the same result with the brosylate (*J. Amer. Chem. Soc.*, 92, 3496 (1970)) and, in addition provided a detailed product analysis. We thank Professor Nickon for informing us of his results prior to publication. Also see W. Kirmse, G. Arend, and R. Siegfried, *Angew. Chem., Int. Ed. Engl.*, 9, 165 (1970).

Table II. *exo/endo* Rate Ratios for Acetolysis of 1-Substituted 2-Norbornyl Tosylates, 25°

Substituent	k_{exo}/k_{endo}	Ref
CN[a]	3[a]	*b*
H	300	*c*
OCH_3	6,200	This work
CH_3	14,000	*c*
p-$NO_2C_6H_4$	94	*d*
p-ClC_6H_4	460	*d*
C_6H_5	1,150	*d*
p-$CH_3C_6H_4$	1,910	*d*
p-$CH_3OC_6H_4$	2,270	*d*

[a] Data for the apobornyl derivatives. [b] Reference 20. [c] Reference 15. [d] Reference 13.

delocalization to the 1 position (alternatively, it has been suggested that the different dipolar orientation may be responsible).[23] However, in comparison with the rate of the parent compound, 1-methoxy substitution produced practically no rate enhancement (a factor of 1.2 at 25°) for *exo* solvolysis. This means that the +R carbonium ion stabilizing ability of the methoxy group has just overcome its large −I inductive effect.[24]

We conclude that methoxy groups have serious limitations as probes for charge delocalization in carbonium ions, *especially in saturated systems*. When little p character is developed at the methoxy-bearing carbon in the transition state resonance involving the oxygen lone pair electrons is not very effective, and the adverse inductive effect of the methoxy substituent may dominate the situation.[25] In such saturated systems, methyl groups (+R *and* +I) may be more effective. A different situation exists when methoxy is attached to a carbon (unsaturated or cyclopropanoid) with a large amount of p character. In such cases developing positive charge is transmitted relatively efficiently to oxygen, the +R effect dominates, and large rate enhancements are observed.[4-7]

Acknowledgments. This research was supported by grants from the National Science Foundation, the National Institutes of Health (AI 07766), and the Petroleum Research Fund, administered by the American Chemical Society.

(23) Reference 20, footnote 11.
(24) *Cf.* F. R. Jensen and B. E. Smart, *J. Amer. Chem. Soc.*, **91**, 5688 (1969).
(25) For an additional recent example, see I. Lillien and L. Handloser, *Tetrahedron Lett.*, 1213 (1970).
(26) National Institutes of Health Postdoctoral Fellows: (a) 1967–1968; (b) 1968–1970.

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Received April 20, 1970

Atomic Coordinates of Optically Active Pyrrolid-2-ones

Sir:

Owing to the importance of substituted pyrrolid-2-one molecular systems as models for optical rotation studies on the peptide chromophore,[1-8] we have initiated a program of determining the crystal structures of a series of these compounds. In the present communication we report the atomic coordinates for L-5-iodomethylpyrrolid-2-one and L-pyrrolid-2-one-5-carboxamide. These coordinates are the first to appear for pyrrolid-2-ones. They are particularly significant for optical rotation studies because of the high,[2-4] and perhaps even extreme,[6] sensitivity of calculated rotational strengths to the atomic parameters of moieties near the acyl portion of the peptide. A small but consistent nonplanarity, which should not be neglected for members of the molecular series with low rotational strength, such as the L-3-aminopyrrolid-2-one,[1,2,5,6] has been found. Furthermore, it has recently been suggested that the sign of the rotational strength of the peptide n–π^* transition in lactams depends on the skeletal geometry and not on the peripheral molecular asymmetry.[9] The results, reported here, demonstrate that this is not the case, since both lactams have the same chiral sense or skeletal geometry but have large and oppositely signed rotational strengths (-35×10^{-40} for L-5-iodomethylpyrrolid-2-one and $+9.4 \times 10^{-40}$ for L-pyrrolid-2-one-5-carboxamide).[10] In addition these results confirm the absolute configuration of the series of pyrrolid-2-ones[2] derived initially from cyclization of glutamic acid.

L-5-Iodomethylpyrrolid-2-one crystallizes in the space group $P2_12_12_1$ with four molecules in the unit cell of $a = 10.45$ (1), $b = 9.64$ (1), $c = 6.929$ (7) Å. A crystal $0.5 \times 0.2 \times 0.1$ mm was mounted along the needle axis on a Pailred diffractometer. For layers 0–7, 943 intensities above background were collected with Mo Kα radiation and a quartz monochromator. No correction for absorption was included. The structure was solved by Patterson and Fourier methods. Application of the *R*-factor ratio test[11] indicated the absolute configuration with a probable error of less than 0.5%. After six peaks at reasonable locations for hydrogen atoms appeared on a difference map (the hydrogens attached to C_1 did not appear), all hydrogen atoms were assigned tetrahedral positions 1.08 Å from the carbon atoms and a planar location 1.03 Å from the nitrogen atom. Full-matrix, least-squares refinement based on anisotropic temperature factors for the iodine atom and fixed hydrogen parameters led to final weighted and unweighted *R* factors of 6.3% and 5.1%. The positional parameters with their standard deviations from this analysis are listed in Table I and the bond distances and angles with their standard deviations in Figure 1. The five-mem-

Table I. Final Atomic Parameters for Iodomethylpyrrolid-2-one

	x	*y*	*z*	*B*,[a] Å²
I	0.1396 (1)	0.0704 (1)	1.0813 (2)	
O	0.304 (1)	0.461 (1)	0.687 (2)	4.7 (2)
N_1	0.130 (1)	0.385 (1)	0.858 (1)	3.6 (2)
C_2	0.206 (1)	0.389 (1)	0.700 (2)	3.3 (2)
C_3	0.156 (1)	0.298 (1)	5.549 (2)	4.3 (2)
C_4	0.046 (1)	0.220 (2)	0.643 (2)	5.0 (3)
C_5	0.018 (1)	0.298 (1)	0.836 (2)	4.0 (2)
C_6	−0.099 (1)	0.215 (2)	1.007 (2)	4.3 (3)
H_1	0.151	0.438	0.983	4.5
H_2	0.228	0.228	0.498	4.5
H_3	0.121	0.360	0.428	4.5
H_4	−0.039	0.220	0.550	4.5
H_5	0.072	0.113	0.671	4.5
H_6	−0.064	0.366	0.811	4.5
H_7	−0.097	0.158	0.983	4.5
H_8	−0.024	0.286	1.128	4.5

[a] The anisotropic thermal motion is expressed in the form exp $[-(\beta_{11}h^2 + \beta_{22}k^2 + \beta_{33}l^2 + 2\beta_{12}hk + 2\beta_{13}hl + 2\beta_{23}kl)]$ Å², where $\beta_{11} = 0.0112$ (1), $\beta_{22} = 0.0107$ (1), $\beta_{33} = 0.0337$ (3), $\beta_{12} = 0.0014$ (1), $\beta_{13} = 0.0048$ (1), and $\beta_{23} = 0.0003$ (2).

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