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STRUCTURE OF "DIMETHYLENETRICYCLOHEXANONE"

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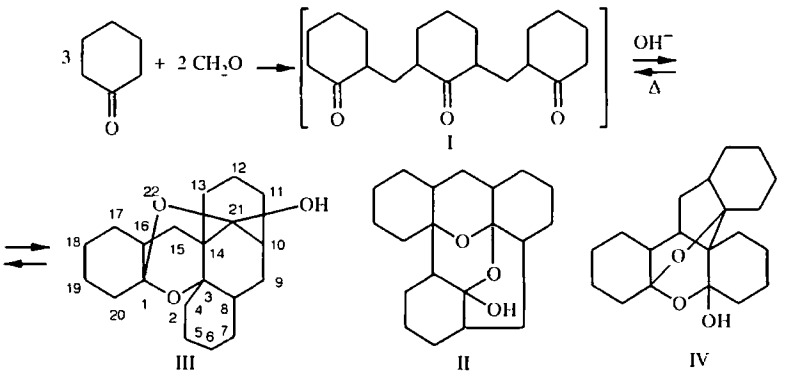
The structure of the product of condensation in basic medium of two molecules of formaldehyde with three molecules of cyclohexanone has been proved using X-ray structural analysis.

The product of condensation in basic medium of two molecules of formaldehyde with three molecules of cyclohexanone ("dimethylenetricyclohexanone") was first prepared and its composition shown to be $C_{20}H_{30}O_3$ by M. N. Tilichenko [1]. By an IR study of this compound it was shown not to be the triketone I, as might have been expected from the diketone condensation scheme proposed by him [2]. The spectrum showed the absence of carbonyl group absorption and the presence of absorption for the OH group. On this basis, and by analogy with the properties of a series of substituted δ-bicyclanones, formula II was proposed for the compound. It was shown that heating above the melting point caused decyclization to triketone I and that the latter was converted quantitatively to the starting compound by the action of an alcoholic solution of base.



A more recent study [3] based on the analysis of ^{13}C NMR spectra and the study of the reaction of intermediate products in the "dimethylenetricyclohexanone" conversion has led to the proposal that the structure is 21-hydroxy-2,22-dioxahexacyclo[12,6,2,$0^{1,16}$,$0^{3,8}$,$0^{3,14}$,$0^{11,22}$]docosane (III).

However, there has been a recent report [4] describing the product of cyclohexanone and paraformaldehyde condensation in a base medium, which coincides in melting point and ^{13}C spectrum with compound III, but for which structure IV was proposed (not the same as structure III). We have repeated the method [4] and found that the material so obtained does not show a depressed melting point upon mixed melting with III and that the IR and ^{13}C NMR spectra coincide. In order to eliminate this contradiction we have turned to an X-ray structural analysis which has uniquely shown that the structure is, in fact, III. The overall view of the molecule is given in Fig. 1 and bond lengths, valence and torsional angles are given in Tables 1, 2, and 3 respectively.

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TABLE 1. Bond Lengths (d) in the Molecule of Compound III.

Bond	d, Å	Bond	d, Å
$O_{(1)}$–$C_{(1)}$	1.415(2)	$C_{(8)}$–$C_{(9)}$	1.543(2)
$O_{(1)}$–$C_{(19)}$	1.462(2)	$C_{(8)}$–$C_{(18)}$	1.561(2)
$O_{(2)}$–$C_{(1)}$	1.436(2)	$C_{(9)}$–$C_{(10)}$	1.524(3)
$O_{(2)}$–$C_{(20)}$	1.443(2)	$C_{(10)}$–$C_{(11)}$	1.527(3)
$O_{(3)}$–$C_{(20)}$	1.404(2)	$C_{(11)}$–$C_{(12)}$	1.535(3)
$C_{(1)}$–$C_{(2)}$	1.511(2)	$C_{(12)}$–$C_{(13)}$	1.530(3)
$C_{(1)}$–$C_{(6)}$	1.523(2)	$C_{(12)}$–$C_{(20)}$	1.530(2)
$C_{(2)}$–$C_{(3)}$	1.525(3)	$C_{(13)}$–$C_{(14)}$	1.522(2)
$C_{(3)}$–$C_{(4)}$	1.514(4)	$C_{(14)}$–$C_{(15)}$	1.529(2)
$C_{(4)}$–$C_{(5)}$	1.522(4)	$C_{(14)}$–$C_{(19)}$	1.543(2)
$C_{(5)}$–$C_{(6)}$	1.530(2)	$C_{(15)}$–$C_{(16)}$	1.516(3)
$C_{(6)}$–$C_{(7)}$	1.534(3)	$C_{(16)}$–$C_{(17)}$	1.513(3)
$C_{(7)}$–$C_{(8)}$	1.542(2)	$C_{(17)}$–$C_{(18)}$	1.530(2)
$C_{(8)}$–$C_{(20)}$	1.532(2)	$C_{(18)}$–$C_{(19)}$	1.532(2)

TABLE 2. Valence Angles (ω) in the Molecule of Compound III

Angle	ω, deg.	Angle	ω, deg.
$C_{(1)}$–$O_{(1)}$–$C_{(19)}$	114.70(10)	$C_{(10)}$–$C_{(11)}$–$C_{(12)}$	113.9(2)
$C_{(1)}$–$O_{(2)}$–$C_{(20)}$	113.18(11)	$C_{(13)}$–$C_{(12)}$–$C_{(20)}$	109.19(13)
$O_{(1)}$–$C_{(1)}$–$O_{(2)}$	108.54(11)	$C_{(13)}$–$C_{(12)}$–$C_{(11)}$	115.0(2)
$O_{(1)}$–$C_{(1)}$–$C_{(2)}$	107.46(12)	$C_{(20)}$–$C_{(12)}$–$C_{(11)}$	108.6(2)
$O_{(2)}$–$C_{(1)}$–$C_{(2)}$	107.23(13)	$C_{(14)}$–$C_{(13)}$–$C_{(12)}$	114.9(2)
$O_{(1)}$–$C_{(1)}$–$C_{(6)}$	110.02(12)	$C_{(13)}$–$C_{(14)}$–$C_{(15)}$	112.0(2)
$O_{(2)}$–$C_{(1)}$–$C_{(6)}$	110.0(12)	$C_{(13)}$–$C_{(14)}$–$C_{(19)}$	113.00(13)
$C_{(2)}$–$C_{(1)}$–$C_{(6)}$	113.43(14)	$C_{(15)}$–$C_{(14)}$–$C_{(19)}$	110.15(14)
$C_{(1)}$–$C_{(2)}$–$C_{(3)}$	111.0(2)	$C_{(16)}$–$C_{(15)}$–$C_{(14)}$	111.4(2)
$C_{(4)}$–$C_{(3)}$–$C_{(2)}$	111.3(2)	$C_{(17)}$–$C_{(16)}$–$C_{(15)}$	111.3(2)
$C_{(3)}$–$C_{(4)}$–$C_{(5)}$	111.5(2)	$C_{(16)}$–$C_{(17)}$–$C_{(18)}$	112.3(2)
$C_{(4)}$–$C_{(5)}$–$C_{(6)}$	112.0(2)	$C_{(17)}$–$C_{(18)}$–$C_{(19)}$	112.0(2)
$C_{(1)}$–$C_{(6)}$–$C_{(5)}$	111.01(14)	$O_{(1)}$–$C_{(19)}$–$C_{(18)}$	106.80(12)
$C_{(1)}$–$C_{(6)}$–$C_{(7)}$	107.46(13)	$O_{(1)}$–$C_{(19)}$–$C_{(14)}$	106.74(12)
$C_{(5)}$–$C_{(6)}$–$C_{(7)}$	113.6(2)	$C_{(18)}$–$C_{(19)}$–$C_{(14)}$	108.60(13)
$C_{(6)}$–$C_{(7)}$–$C_{(8)}$	109.95(13)	$O_{(1)}$–$C_{(19)}$–$C_{(8)}$	106.88(11)
$C_{(20)}$–$C_{(8)}$–$C_{(7)}$	107.55(13)	$C_{(18)}$–$C_{(19)}$–$C_{(8)}$	114.09(13)
$C_{(20)}$–$C_{(8)}$–$C_{(9)}$	108.26(12)	$C_{(14)}$–$C_{(19)}$–$C_{(8)}$	113.27(12)
$C_{(7)}$–$C_{(8)}$–$C_{(9)}$	110.32(14)	$O_{(3)}$–$C_{(20)}$–$O_{(2)}$	108.78(11)
$C_{(20)}$–$C_{(8)}$–$C_{(19)}$	106.42(12)	$O_{(3)}$–$C_{(20)}$–$C_{(12)}$	112.41(13)
$C_{(7)}$–$C_{(8)}$–$C_{(19)}$	108.61(12)	$O_{(2)}$–$C_{(20)}$–$C_{(12)}$	107.18(12)
$C_{(9)}$–$C_{(8)}$–$C_{(19)}$	115.35(13)	$O_{(3)}$–$C_{(20)}$–$C_{(8)}$	108.17(12)
$C_{(10)}$–$C_{(9)}$–$C_{(8)}$	116.5(2)	$O_{(2)}$–$C_{(20)}$–$C_{(8)}$	109.22(11)
$C_{(9)}$–$C_{(10)}$–$C_{(11)}$	113.4(2)	$C_{(12)}$–$C_{(20)}$–$C_{(8)}$	111.02(13)

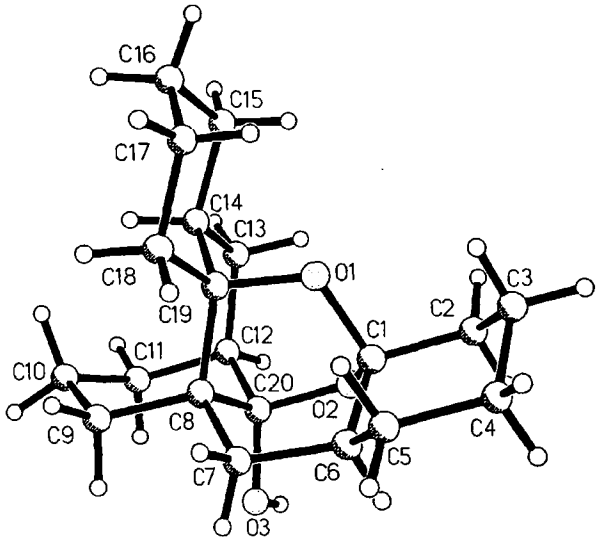

Fig. 1. Overall view of molecule III.

As is apparent from Fig. 1 and Table 4, in the molecule of substance III the tetrahydropyran rings are in a boat conformation and the cyclohexane has a chair conformation. The geometrical parameters for the molecule (bond lengths and valence angles) are standard [5].

In the crystal the intermolecular hydrogen bonds $O_{(3)}$–$H_{(30)}$···$O_{(2)}$ (2-x, -y, 1-z) [$O_{(3)}$···$O_{(2)}$ 2.823 (2), $O_{(3)}$–$H_{(30)}$ 0.83 (2), $H_{(30)}$···$O_{(2)}$ 2.00 (2) Å, $O_{(3)}$–$H_{(30)}$···$O_{(2)}$ angle 167 (2)°] cause the molecules of compound III to exist as dimers (Fig. 2).

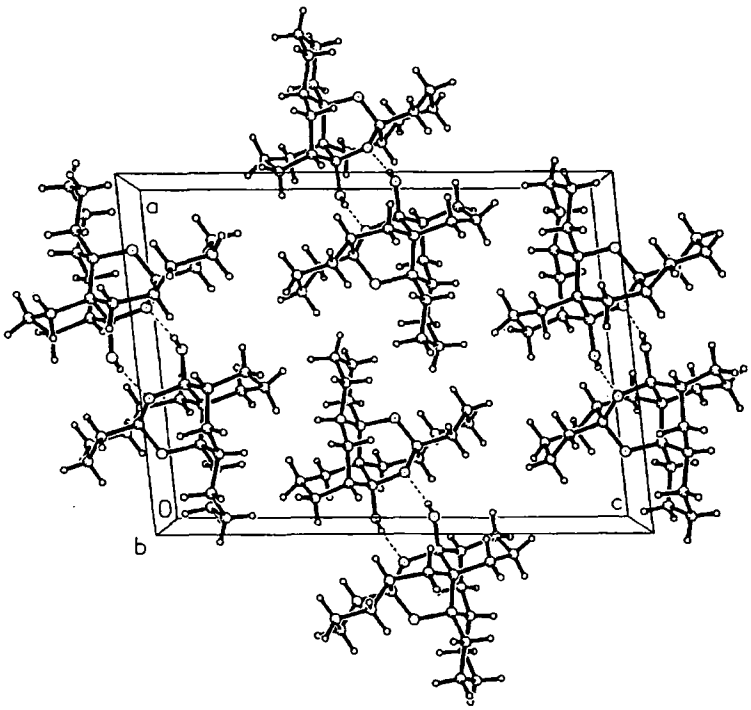

Fig. 2. Projection along *ac* axis of the crystalline structure of III. Broken lines shown the intermolecular hydrogen bonds O–H···O.

TABLE 3. Torsion Angles (τ) in Compound III

Angle	τ, deg.	Angle	τ, deg.
$C_{(19)}$–$O_{(1)}$–$C_{(1)}$–$O_{(2)}$	54.4(1)	$C_{(16)}$–$C_{(17)}$–$C_{(18)}$–$C_{(19)}$	54.0(2)
$C_{(19)}$–$O_{(1)}$–$C_{(1)}$–$C_{(2)}$	170.1(1)	$C_{(1)}$–$O_{(1)}$–$C_{(19)}$–$C_{(18)}$	129.7(1)
$C_{(19)}$–$O_{(1)}$–$C_{(1)}$–$C_{(6)}$	-66.0(1)	$C_{(1)}$–$O_{(1)}$–$C_{(19)}$–$C_{(14)}$	-114.3(1)
$C_{(20)}$–$O_{(2)}$–$C_{(1)}$–$C_{(2)}$	-63.2(1)	$C_{(1)}$–$O_{(1)}$–$C_{(19)}$–$C_{(8)}$	7.2(2)
$C_{(20)}$–$O_{(2)}$–$C_{(1)}$–$C_{(2)}$	-179.0(1)	$C_{(17)}$–$C_{(18)}$–$C_{(19)}$–$O_{(1)}$	58.4(2)
$C_{(20)}$–$O_{(2)}$–$C_{(1)}$–$C_{(6)}$	57.2(1)	$C_{(17)}$–$C_{(18)}$–$C_{(19)}$–$C_{(14)}$	-56.4(2)
$O_{(1)}$–$C_{(1)}$–$C_{(2)}$–$C_{(3)}$	68.1(2)	$C_{(17)}$–$C_{(18)}$–$C_{(19)}$–$C_{(8)}$	176.2(2)
$O_{(2)}$–$C_{(1)}$–$C_{(2)}$–$C_{(3)}$	-175.4(1)	$C_{(13)}$–$C_{(14)}$–$C_{(19)}$–$O_{(1)}$	70.1(2)
$C_{(6)}$–$C_{(1)}$–$C_{(2)}$–$C_{(3)}$	-53.7(2)	$C_{(15)}$–$C_{(14)}$–$C_{(19)}$–$O_{(1)}$	-56.0(2)
$C_{(1)}$–$C_{(2)}$–$C_{(3)}$–$C_{(4)}$	55.1(2)	$C_{(13)}$–$C_{(14)}$–$C_{(19)}$–$C_{(18)}$	-175.1(1)
$C_{(2)}$–$C_{(3)}$–$C_{(4)}$–$C_{(5)}$	-56.2(2)	$C_{(15)}$–$C_{(14)}$–$C_{(19)}$–$C_{(18)}$	58.8(2)
$C_{(3)}$–$C_{(4)}$–$C_{(5)}$–$C_{(6)}$	55.0(2)	$C_{(13)}$–$C_{(14)}$–$C_{(19)}$–$C_{(8)}$	-47.3(2)
$O_{(1)}$–$C_{(1)}$–$C_{(6)}$–$C_{(5)}$	-68.2(2)	$C_{(15)}$–$C_{(14)}$–$C_{(19)}$–$C_{(8)}$	-173.4(1)
$O_{(2)}$–$C_{(1)}$–$C_{(6)}$–$C_{(5)}$	172.3(1)	$C_{(20)}$–$C_{(8)}$–$C_{(19)}$–$O_{(1)}$	-61.0(1)
$C_{(2)}$–$C_{(1)}$–$C_{(6)}$–$C_{(5)}$	52.2(2)	$C_{(7)}$–$C_{(8)}$–$C_{(19)}$–$O_{(1)}$	54.5(1)
$O_{(1)}$–$C_{(1)}$–$C_{(6)}$–$C_{(7)}$	56.6(1)	$C_{(9)}$–$C_{(8)}$–$C_{(19)}$–$O_{(1)}$	178.9(1)
$O_{(2)}$–$C_{(1)}$–$C_{(6)}$–$C_{(7)}$	-62.9(1)	$C_{(20)}$–$C_{(8)}$–$C_{(19)}$–$C_{(18)}$	-178.8(1)
$C_{(2)}$–$C_{(1)}$–$C_{(6)}$–$C_{(7)}$	177.0(1)	$C_{(7)}$–$C_{(8)}$–$C_{(19)}$–$C_{(18)}$	-63.3(2)
$C_{(4)}$–$C_{(5)}$–$C_{(6)}$–$C_{(1)}$	-52.2(2)	$C_{(9)}$–$C_{(8)}$–$C_{(19)}$–$C_{(18)}$	61.1(2)
$C_{(4)}$–$C_{(5)}$–$C_{(6)}$–$C_{(7)}$	-173.4(2)	$C_{(20)}$–$C_{(8)}$–$C_{(19)}$–$C_{(14)}$	56.2(2)
$C_{(1)}$–$C_{(6)}$–$C_{(7)}$–$C_{(8)}$	5.5(2)	$C_{(7)}$–$C_{(8)}$–$C_{(19)}$–$C_{(14)}$	171.8(1)
$C_{(5)}$–$C_{(6)}$–$C_{(7)}$–$C_{(8)}$	128.7(2)	$C_{(9)}$–$C_{(8)}$–$C_{(19)}$–$C_{(14)}$	-63.8(2)
$C_{(6)}$–$C_{(7)}$–$C_{(8)}$–$C_{(20)}$	54.1(2)	$C_{(1)}$–$O_{(2)}$–$C_{(20)}$–$O_{(3)}$	-111.3(1)
$C_{(6)}$–$C_{(7)}$–$C_{(8)}$–$C_{(9)}$	171.9(1)	$C_{(1)}$–$O_{(2)}$–$C_{(20)}$–$C_{(12)}$	126.9(1)
$C_{(6)}$–$C_{(7)}$–$C_{(8)}$–$C_{(19)}$	-60.7(2)	$C_{(1)}$–$O_{(2)}$–$C_{(20)}$–$C_{(8)}$	6.6(1)
$C_{(20)}$–$C_{(8)}$–$C_{(9)}$–$C_{(10)}$	-49.2(2)	$C_{(13)}$–$C_{(12)}$–$C_{(20)}$–$O_{(3)}$	-176.4(1)
$C_{(7)}$–$C_{(8)}$–$C_{(9)}$–$C_{(10)}$	-166.7(1)	$C_{(11)}$–$C_{(12)}$–$C_{(20)}$–$O_{(3)}$	57.6(2)
$C_{(19)}$–$C_{(8)}$–$C_{(9)}$–$C_{(10)}$	69.8(2)	$C_{(13)}$–$C_{(12)}$–$C_{(20)}$–$O_{(2)}$	-56.9(2)
$C_{(8)}$–$C_{(9)}$–$C_{(10)}$–$C_{(11)}$	41.7(2)	$C_{(11)}$–$C_{(12)}$–$C_{(20)}$–$O_{(2)}$	177.1(1)
$C_{(9)}$–$C_{(10)}$–$C_{(11)}$–$C_{(12)}$	-43.8(3)	$C_{(13)}$–$C_{(12)}$–$C_{(20)}$–$C_{(8)}$	62.3(2)
$C_{(10)}$–$C_{(11)}$–$C_{(12)}$–$C_{(13)}$	-68.1(2)	$C_{(11)}$–$C_{(12)}$–$C_{(20)}$–$C_{(8)}$	-63.7(2)
$C_{(10)}$–$C_{(11)}$–$C_{(12)}$–$C_{(20)}$	54.5(2)	$C_{(7)}$–$C_{(8)}$–$C_{(20)}$–$O_{(3)}$	55.6(2)
$C_{(20)}$–$C_{(12)}$–$C_{(13)}$–$C_{(14)}$	-51.2(2)	$C_{(9)}$–$C_{(8)}$–$C_{(20)}$–$O_{(3)}$	-63.5(2)
$C_{(11)}$–$C_{(12)}$–$C_{(13)}$–$C_{(14)}$	71.1(2)	$C_{(19)}$–$C_{(8)}$–$C_{(20)}$–$O_{(3)}$	171.9(1)
$C_{(12)}$–$C_{(13)}$–$C_{(14)}$–$C_{(15)}$	169.5(1)	$C_{(7)}$–$C_{(8)}$–$C_{(20)}$–$O_{(2)}$	-62.6(1)
$C_{(12)}$–$C_{(13)}$–$C_{(14)}$–$C_{(19)}$	44.4(2)	$C_{(9)}$–$C_{(8)}$–$C_{(20)}$–$O_{(2)}$	178.2(1)
$C_{(13)}$–$C_{(14)}$–$C_{(15)}$–$C_{(16)}$	174.1(1)	$C_{(19)}$–$C_{(8)}$–$C_{(20)}$–$O_{(2)}$	53.6(1)
$C_{(19)}$–$C_{(14)}$–$C_{(15)}$–$C_{(16)}$	-59.2(2)	$C_{(7)}$–$C_{(8)}$–$C_{(20)}$–$C_{(12)}$	179.4(1)
$C_{(14)}$–$C_{(15)}$–$C_{(16)}$–$C_{(17)}$	55.3(2)	$C_{(9)}$–$C_{(8)}$–$C_{(20)}$–$C_{(12)}$	60.2(2)
$C_{(15)}$–$C_{(16)}$–$C_{(17)}$–$C_{(18)}$	-52.4(2)	$C_{(19)}$–$C_{(8)}$–$C_{(20)}$–$C_{(12)}$	-64.3(1)

TABLE 4. Coordinates ($\times 10^4$) and Isotropic Equivalent (for Hydrogen Isotropic) Atomic Thermal Parameters in Compound III.

Atom	*x*	*y*	*z*	*U*
1	2	3	4	5
$O_{(1)}$	7044(1)	2066(1)	4961(1)	30(1)
$O_{(2)}$	8686(1)	895(1)	4877(1)	31(1)
$O_{(3)}$	10119(1)	1957(2)	5636(1)	41(1)
$C_{(1)}$	7911(1)	2116(2)	4538(1)	30(1)
$C_{(2)}$	7559(1)	1401(3)	3759(1)	44(1)
$C_{(3)}$	6796(2)	2697(3)	3329(1)	58(1)
$C_{(4)}$	7251(2)	4577(4)	3296(1)	75(1)
$C_{(5)}$	7592(2)	5305(3)	4082(1)	59(1)
$C_{(6)}$	8349(1)	4026(2)	4535(1)	38(1)

TABLE 4 (continued)

1	2	3	4	5
$C_{(7)}$	8615(1)	4601(2)	5360(1)	40(1)
$C_{(8)}$	8450(1)	3001(2)	5889(1)	31(1)
$C_{(9)}$	8863(1)	3477(3)	6709(1)	49(1)
$C_{(10)}$	8981(2)	1913(3)	7268(1)	59(1)
$C_{(11)}$	9436(2)	212(3)	6945(1)	57(1)
$C_{(12)}$	8970(1)	-249(2)	6139(1)	39(1)
$C_{(13)}$	7831(1)	-842(2)	6068(1)	43(1)
$C_{(14)}$	7058(1)	658(2)	6184(1)	37(1)
$C_{(15)}$	5936(1)	61(3)	5964(1)	48(1)
$C_{(16)}$	5171(1)	1497(3)	6158(1)	58(1)
$C_{(17)}$	5391(1)	3291(3)	5799(1)	52(1)
$C_{(18)}$	6524(1)	3878(3)	5977(1)	42(1)
$C_{(19)}$	7285(1)	2421(2)	5768(1)	29(1)
$C_{(20)}$	9081(1)	1401(2)	5636(1)	31(1)
$H_{(30)}$	10451(15)	1029(26)	5547(10)	46(5)
$H_{(21)}$	7271(15)	155(29)	3822(11)	57(6)
$H_{(22)}$	8155(15)	1336(24)	3496(10)	45(5)
$H_{(31)}$	6578(16)	2187(27)	2819(13)	64(6)
$H_{(32)}$	6171(17)	2798(27)	3587(11)	57(6)
$H_{(41)}$	6756(19)	5470(33)	3036(13)	80(7)
$H_{(42)}$	7868(19)	4490(31)	3009(14)	77(7)
$H_{(51)}$	7880(18)	6488(32)	4075(12)	73(7)
$H_{(52)}$	6960(17)	5446(27)	4367(12)	58(6)
$H_{(61)}$	8982(14)	3946(22)	4288(9)	37(4)
$H_{(71)}$	8195(16)	5671(27)	5490(11)	58(6)
$H_{(72)}$	9322(15)	5003(26)	5461(10)	48(5)
$H_{(91)}$	8443(16)	4462(29)	6882(12)	64(6)
$H_{(92)}$	9559(16)	4003(27)	6670(11)	60(6)
$H_{(101)}$	9461(17)	2262(28)	7709(13)	65(6)
$H_{(102)}$	8306(16)	1603(25)	7473(10)	51(5)
$H_{(111)}$	9358(17)	-813(31)	7286(13)	70(7)
$H_{(112)}$	10165(18)	375(29)	6941(12)	63(6)
$H_{(12)}$	9347(13)	-1256(24)	5957(9)	41(5)
$H_{(131)}$	7742(16)	-1856(28)	6417(11)	59(6)
$H_{(132)}$	7635(15)	-1422(26)	5540(12)	56(5)
$H_{(14)}$	7077(14)	1009(24)	6700(11)	46(5)
$H_{(151)}$	5831(16)	-1146(29)	6196(11)	60(6)
$H_{(152)}$	5858(14)	-253(27)	5424(12)	54(5)
$H_{(161)}$	4481(17)	1079(27)	5981(11)	60(6)
$H_{(162)}$	5227(17)	1613(29)	6708(13)	69(6)
$H_{(171)}$	4978(17)	4333(31)	5944(12)	69(6)
$H_{(172)}$	5230(15)	3156(26)	5263(12)	57(6)
$H_{(181)}$	6630(14)	5069(28)	5716(11)	51(5)
$H_{(182)}$	6651(15)	4127(26)	6522(12)	55(6)

EXPERIMENTAL

X-Ray Structural Investigation of Compound III. Colorless crystals of compound III ($C_{20}H_{30}O_3$) are monoclinic, at 25°C: a = 12.936 (6), b = 7.400 (4), c = 17.807 (9) Å, β = 95.96 (4)°, V = 1696 (2) Å^3, d_{calc} = 1.247 g/cm^3, Z = 4, space group $P2(1)/n$. Unit cell parameters and intensities of 3471 reflections were measured on a Siemens P3/Pc, four circle automatic diffractometer (λMoKa graphite monochromator, $\theta/2$ scanning, θ_{max} = 27°).The structure was solved by a direct method and refined in a full matrix, least squares analysis in the anisotropic approximation for nonhydrogen atoms. Hydrogen atoms were localized directly by difference Fourier synthesis

and refined in the isotropic approximation. Final values of the difference factors were $R1$ = 0.043 for 2436 independent reflections. All calculations were carried out using the SHELXTL PLUS and SHELXL-93 programs. Coordinates and isotropic (equivalent for non-hydrogen atoms) atomic thermal parameters are given in Table 4.

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