

mixture indicated 62% acetate **28**, 19% alcohol **27** (transesterification product), and 19% methyl ether **29** (solvolysis product).

Preparation of 5a,11a-Diacetoxyjanusene (19).—To a solution of 218 mg (0.48 mmol) of 5a-hydroxy-11a-acetoxyjanusene (**8**) in 15 ml of acetic anhydride was added 6 drops of concentrated sulfuric acid. The mixture was stirred at 80° for 15 min and then worked up. The crude product, 265 mg (110%), was identified by its pmr spectrum as 5a,11a-diacetoxyjanusene (**19**). Diacetate **19** was crystallized from methanol: mp 270.5–272°; pmr (CDCl₃) τ 8.40 (s, 6, OAc), 4.48 (s, 4), 2.85–3.40 (m, 16, aromatics).

Anal. Calcd for C₂₄H₂₀O₄: C, 81.93; H, 5.22. Found: C, 81.77; H, 5.22.

Preparation of 5a,11a-Dihydroxyjanusene.—A solution of 199 mg (0.44 mmol) of hydroxyacetate **8** in 25 ml of sodium methoxide-methanol solution (prepared by treating 100 ml of methanol with 0.25 g of sodium) was stirred at reflux for 2 days. The crude product, 155 mg (85%), was identified by its pmr spectrum as 5a,11a-dihydroxyjanusene and it was crystallized

from acetone–95% EtOH: mp >340°; pmr (CDCl₃) τ 8.02 (s, 2, OH), 5.57 (s, 4), 2.87–3.27 (m, 16, aromatics).

Anal. Calcd for C₂₀H₂₂O₂: C, 86.96; H, 5.31. Found: C, 87.17; H, 5.36.

Registry No.—**3**, 29246-46-6; **4**, 29246-47-7; **7**, 29246-48-8; **19**, 29320-07-8; **20**, 29435-62-9; **25**, 29179-05-3; **26**, 29246-49-9; **27**, 29179-06-4; **29**, 29179-07-5; *exo*-6,*endo*-12-dihydroxy-*cis*-isojanusene, 29179-08-6; 6,12-dihydroxy-*trans*-isojanusene, 29179-09-7; 5a,11a-dihydroxyjanusene, 29246-50-2.

Acknowledgments.—The authors are indebted to the National Science Foundation and to the Institute of General Medical Sciences (Public Health Service Grant GM-12139) for support of this work.

Bridged Polycyclic Compounds. LXVIII. The Proton Magnetic Resonance Spectra of Some Derivatives of Janusene, Hemiisojanusene, and Isojanusene¹

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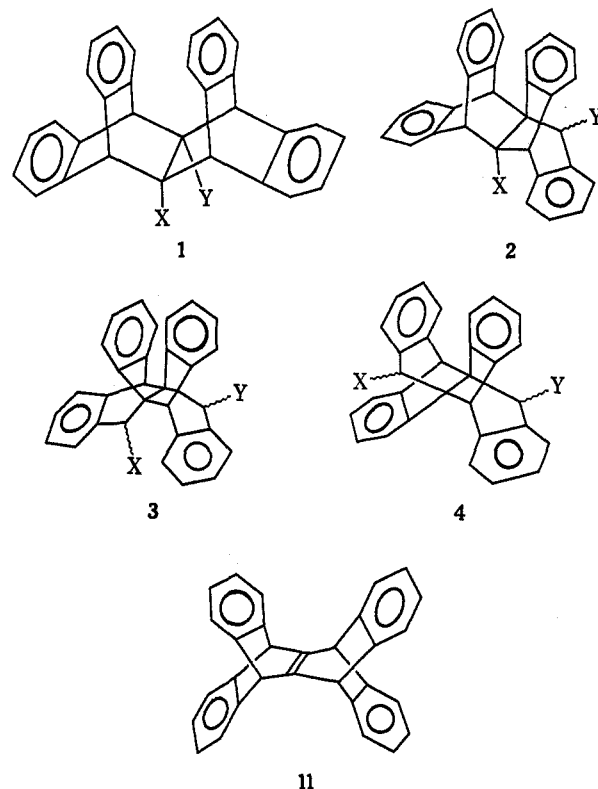
Received August 6, 1970

Proton magnetic resonance spectra are given for 41 compounds of various polyhydrodi-*o*-benzenonaphthalene types. Based upon correlations, it is possible to assign structures to many derivatives of janusene, hemiisojanusene, *cis*-isojanusene, and *trans*-isojanusene.

The examination of the carbonium ion reactions of janusene, hemiisojanusene, and isojanusene derivatives^{1–4} was made possible by interpretation of pmr spectra. Independent syntheses of most of these compounds was not practicable. However, these spectra, when coupled with certain specific reactions and consideration of possible isomeric structures, appear quite conclusive in making structure assignments.

All spectra were obtained using a Varian A-60A nuclear magnetic resonance instrument. The spectra were taken in deuteriochloroform, usually as saturated solutions, and were scanned over τ 1.7–10.0 using tetramethylsilane (τ 10.00) as an internal standard.

Generally, the pmr spectra of disubstituted janusenes (**1**), hemiisojanusenes (**2**), *cis*-isojanusenes (**3**), and *trans*-isojanusenes (**4**) consist of a complex multiplet centered approximately at τ 3.0, which corresponds to the aromatic hydrogens, and a series of singlets, which arise from the aliphatic hydrogens.⁵ Although the general patterns of the singlets are such that skeletal isomers can be easily distinguished, individual proton assignments are difficult and have to be made strictly on the basis of chemical shift data. A number of monosubstituted janusenes (Figure 1), whose structures were derived from chemical knowledge, were prepared as model compounds. In these cases individual proton assignments can be made with certainty, based upon the splitting patterns and expected chemical shifts. The spectral data are listed in Table I.



(1) Previous paper: LXVII. S. J. Cristol and M. A. Imhoff, *J. Org. Chem.*, **36**, 1854 (1971).

(2) S. J. Cristol and M. A. Imhoff, *ibid.*, **36**, 1849 (1971). Methods for trivial nomenclature are given in this paper.

(3) S. J. Cristol, M. A. Imhoff, and D. C. Lewis, *ibid.*, **35**, 1722 (1970).

(4) W. M. Macintyre, M. A. Imhoff, and S. J. Cristol, *ibid.*, **36**, 1865 (1971).

(5) The secondary benzylic protons in **2**, **3**, and **4** were split when the substituent was hydroxyl.

5a-Bromojanusene (**6**), which was prepared by the addition of hydrogen bromide to dehydrojanusene (**11**),³ could be converted back to starting olefin upon treatment with potassium *tert*-butoxide. This same monobromide was also prepared from the radical bromination of janusene (**5**).³ Also, 5a-chlorojanusene (**7**) was prepared by either addition of hydrogen chloride to olefin **11**³ or as a Diels–Alder adduct from the reaction of

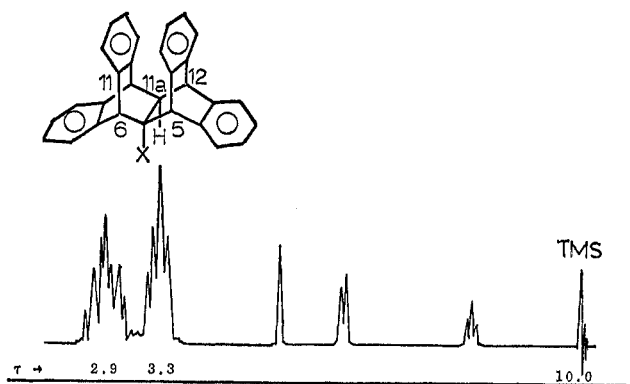


Figure 1.—Structure and general pmr spectrum of a monosubstituted janusene. Omitted from spectrum is absorption, if any, from the substituent.

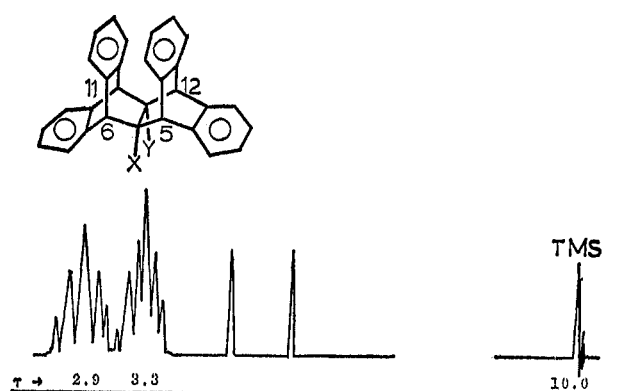


Figure 2.—Structure of a disubstituted janusene when $X \neq Y$ and a typical pmr spectrum. Omitted from the spectrum are proton absorptions contained in X or Y.

TABLE I
PROTON ASSIGNMENTS IN MONOSUBSTITUTED JANUSENES^a

Compd	X	Chemical shifts, τ				J , Hz ^b
		5-H, 6-H	11-H, 12-H	11a-H	Other	
5	H	5.81	5.81 (2)	7.53 (3)		
6	Br	5.23	5.74 (2)	6.79 (3)		2
7	Cl	5.43	5.77 (2)	7.04 (3)		2
8	OH	5.72	5.82 (2)	7.87 (3)	8.48	2
9	OCH ₃	5.35	5.75 (2)	7.70 (3)	6.80	2
10	OAc	4.59	5.74 (2)	7.59 (3)	8.52	2

^a Values are for centers of resonance patterns and are measured in deuteriochloroform. Integral numbers in parentheses after chemical shift values indicate the complexity of the resonance pattern. ^b Coupling between protons at C-11 and C-12 with C-11a.

anthracene with 7-chlorodibenzobicyclo[2.2.2]octatriene⁶ and could be converted to dehydrojanusene (11) upon treatment with base. Together, these reactions support the structures indicated in Figure 1 and Table I.

Also shown in Figure 1 is a typical pmr spectrum of a monosubstituted janusene.⁶ The singlet absorption is assigned to the protons at C-5 and C-6, the doublet to the hydrogens at C-11 and C-12, and the triplet to the proton at C-11a. Omitted from the spectrum is the singlet absorption when the substituent was either acetate, hydroxyl, or methyl ether.

The data in Table I indicate that the benzylic protons at C-5 and C-6 are deshielded by substituent X in the order OAc > Br > OMe > Cl > OH > H. In these compounds the dihedral angle between the carbon-hydrogen bond and the carbon-substituent bond is about 90°. The relative order of these chemical shifts was used in assigning β hydrogens in disubstituted janusenes and in analyzing various reaction mixtures.

Disubstituted Janusenes.—In the disubstituted janusenes discussed here, the functional groups are located

(6) S. J. Cristol and D. C. Lewis, *J. Amer. Chem. Soc.*, **89**, 1476 (1967).

(7) The relative order of deshielding by these substituents is in an order similar to that observed in 7-substituted dibenzobicyclo[2.2.2]octadienes.⁸ A different order of deshielding is observed in the chemical shift data of the hydrogens at C-11a where the dihedral angle is about 0°. In these examples the order is Br > Cl > H > OAc > OMe > OH. Here a shielding property from a diamagnetic anisotropic effect presumably masks the deshielding inductive effect of these substituents.⁹

(8) S. J. Cristol, T. W. Russell, J. R. Mohrig, and D. E. Plorde, *J. Org. Chem.*, **31**, 581 (1966).

(9) J. W. Emsley, J. Feeney, and L. Sutcliffe, "High Resolution Nuclear Magnetic Resonance," Vol. 2, Pergamon Press, Long Island City, N. Y., 1966, p 672.

at C-5a and C-11a.¹⁰ As indicated by the structure in Figure 2,¹¹ there are no adjacent hydrogens, and therefore one would expect only singlets in the aliphatic portion of the spectrum. Also, one may anticipate that the chemical shifts of these absorptions should be similar to those observed for the corresponding singlets in the model compounds. This is, in fact, observed. Figure 2 shows a typical pmr spectrum of a disubstituted janusene, which consists of a complex aromatic proton absorption and a number of singlets depending upon the nature of substituents X and Y. The relative area of the singlets from the bridgehead protons is one-fourth that of the complex multiplet (aromatic protons). When the chemical shifts of the singlets are similar, as in 5a-chloro-11a-methoxyjanusene (18), the proton assignments are based upon the relative order of deshielding by the substituents, X and Y, as determined from the model compounds. In other words, a methoxy group deshields the benzylic protons more than a chloro substituent in the model compounds, and, therefore, the downfield singlet in 18 is assigned to the hydrogens at C-11 and C-12. The assignments are listed in Table II.

TABLE II
PROTON ASSIGNMENTS IN THE DISUBSTITUTED JANUSENE SYSTEM

Compd	X	Y	Chemical shifts, τ		
			5-H, 6-H	11-H, 12-H	Other
12	Br	Br	5.13	5.13	
13	Br	OAc	5.13	4.52	8.32
14	Br	OMe	5.17	5.31	6.53
15	Br	Cl	5.15	5.35	
16	Cl	Cl	5.35	5.35	
17	Cl	OAc	5.36	4.49	8.34
18	Cl	OMe	5.38	5.31	6.56
19	Cl	OH	5.33	5.57	7.77
20	OH	OH	5.57	5.57	8.02
21	OH	OMe	5.57	5.34	6.65 (OMe)
22	OH	OAc	5.56	4.53	8.38 (OAc)
23	OAc	OAc	4.48	4.48	8.40
24	Epoxide		5.32	5.32	
11	Dehydrojanusene		4.87	4.87	

Hemiisojanusene.—All of the isolated derivatives of hemiisojanusene were substituted at C-5a and C-12

(10) Other disubstituted janusenes, in which the substituents were located in the aromatic rings, have been reported by S. J. Cristol and D. C. Lewis.⁶

(11) The structure of 5a,11a-dibromojanusene (12) was confirmed through X-ray analysis by W. M. Macintyre and A. Tench, private communication.

TABLE III
PROTON ASSIGNMENTS IN THE HEMIISOJANUSENE SYSTEM

Compd	X	Y	Chemical shifts, τ				Other
			5-H	6-H	11-H	12-H	
25	Cl	Cl	5.58	5.36	5.00	4.34	
26	Cl	OAc	5.57	5.35	4.97	3.48	7.96
27	Cl	OMe	5.64	5.36	5.06	5.27	6.07
28	OH	OH	5.87	5.55	5.03	5.03	7.65, 6.8
29	OH	OMe	5.76	5.41	5.10	5.26	6.26 (OMe), 6.97 (OH)
30	OH	OAc	5.73	5.37	5.02	3.42	8.06 (OAc)
31	OAc	OAc	4.99	4.67	4.99	3.48	7.98, 8.87
32	OAc	OH	4.92	4.67	5.04	4.91	8.89 (OAc), 7.61 (OH)
33	Br	OAc	5.45	5.17	4.96	? ^a	7.97
34	OH	Keto	5.56	4.39	4.59		8.33

^a Proton absorption was probably buried in the aromatic proton multiplet.

(Figure 3). Inspection of the proposed structures indicated that there are no equivalent protons. Also, none of the aliphatic hydrogens are on adjacent carbons, and therefore only singlets are expected. The observed spectra are consistent with the proposed structures and, in general, consist of a complex multiplet from the aromatic hydrogens, four singlet absorptions (each had a relative area of one-sixteenth that of the complex multiplet), and other singlets if the substituents have hydrogens.

Again, as in the case of disubstituted janusenes, the proton assignments are based upon chemical shifts. Scrutiny of the data in Table III reveals that each spectrum contains an absorption between τ 4.9 and 5.1. This singlet was assigned to the hydrogen at C-11, because it is the one which should be least affected by substituents at C-12 and C-5a. The proton at C-12 is relatively easy to assign due to its characteristic chemical shift which is determined by the deshielding ability of the α substituent. The remaining two singlets are assigned to the hydrogens at C-6 and C-5. Since the environment of the hydrogen at C-6 closely resembled that of the corresponding position in the monosubstituted and disubstituted janusenes, we assign to it the absorption with the more similar chemical shift. This results in the assignment of the lower field singlet to C-6 in all cases. This assignment is further supported when compared to those made for the dibenzobicyclo-[3.2.1]octadiene¹² systems. The proton at C-1 in the [2.2.2] system has a lower chemical shift than the hydrogen at C-1 in the corresponding [3.2.1] system. The hydrogen at C-5 in the hemiisojanusene system is viewed as analogous to the one at C-1 in the [3.2.1] system and, therefore, is expected to appear upfield of the hydrogen at C-6.

Added information about the structure of hemiisojanusenes was obtained from the chemical shift data of the substituents which had hydrogens. This is exemplified by diacetate **31**, which has acetate methyl absorptions at τ 7.98 and 8.87. This latter absorption is higher than normal,^{8,12} and examination of Fieser models of diacetate **31** indicates that the substituent at C-5a is positioned in the "shielding cone" of a benzene ring and, therefore, may be expected to be atypically upfield. This assignment was confirmed by comparing hydroxyacetates **30** and **32**.^{1,2} In hydroxyacetate **30** the acetoxy substituent at C-5a is replaced by a hydroxyl group, and in the pmr spectrum of **30** only the

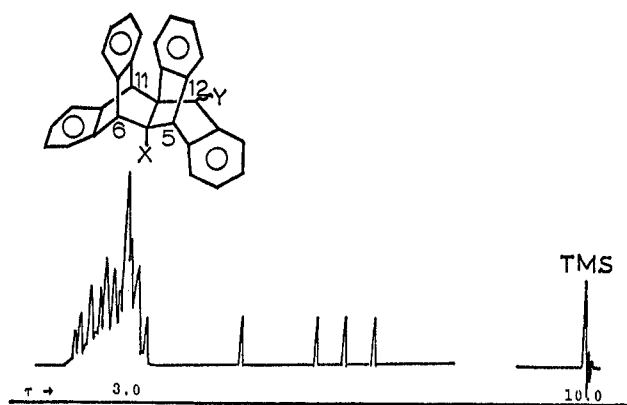
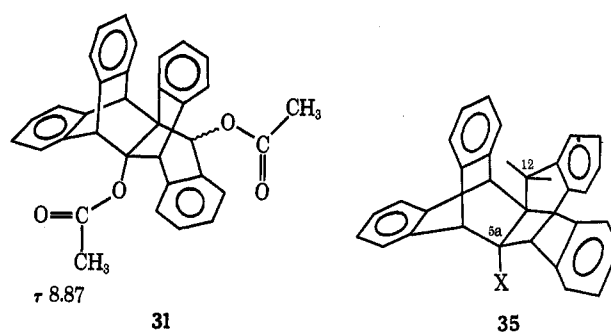


Figure 3.—Structure of a disubstituted hemiisojanusene and a general pmr spectrum. Omitted from the spectrum are the proton absorptions contained in X or Y.

downfield acetate methyl absorption is present. Conversely, the pmr spectrum of hydroxyacetate **32**, in which the acetate at C-12 is substituted by hydroxyl, contains only the upfield acetate methyl absorption.

An alternative structure for hemiisojanusene, which would also contain four unequivalent aliphatic hydrogens, is **35**. In this structure a benzene ring is



located syn to the substituents at C-12. If this were the case, one would expect shielding of the substituents at C-12, but this is not observed. Also, examination of molecular models suggests that the substituents at C-5a should not be greatly shielded. Finally, anti migration, which would give hemiisojanusene **2**, is the preferred direction of rearrangement in similar systems.¹³ Thus it seems certain that hemiisojanusene has a structure resembling **2** and not **35**.

(13) (a) S. J. Cristol, F. P. Parungo, D. E. Florde, and K. Schwarzenbach, *J. Amer. Chem. Soc.*, **87**, 2879 (1965); (b) S. J. Cristol, F. P. Parungo, and D. E. Florde, *ibid.*, **87**, 2870 (1965); (c) S. J. Cristol, R. P. Arganbright, and D. D. Tanner, *J. Org. Chem.*, **28**, 1374 (1963).

TABLE IV
 PROTON ASSIGNMENTS FOR *cis*-ISOJANUENE SYSTEM

Compd	X	C-6 ^a	C-12 ^a	Chemical shifts, τ				
				6-H	12-H	5-H	11-H	Other
36	OAc	exo	endo	3.63	3.92	5.51	5.51	7.89, 8.80
37	OAc	endo	endo	3.92	3.92	5.56	5.56	8.00
38	OH	exo	endo	4.97	5.02	5.34	5.43 ^b	
39	OH	endo	endo	5.01	5.01	5.47	5.47	
40	Keto					4.96	4.96	

^a Configuration of substituent, X; configuration of the proton is opposite. ^b Singlet at τ 5.43 assigned to 11-H since the chemical shift resembles τ 5.47 in the diendo compound **39**.

 TABLE V
 PROTON ASSIGNMENTS IN DISUBSTITUTED *trans*-ISOJANUENES

Compd	X	C-6 ^a	C-12 ^a	Chemical shifts, τ				
				6-H	12-H	5-H	11-H	Other
41	OAc	exo	endo	3.60	3.80	5.44	5.16 ^b	7.72, 8.27
42	OAc	endo	endo	3.85	3.85	5.24	5.24	8.20
44	OH	endo	endo	5.16	5.16	5.50	5.50	
45	Keto					4.97	4.97	

^a Configuration of substituent, X; configuration of the proton is opposite. ^b Singlet assigned to 11-H because the chemical shift resembles τ 5.24 (more closely than τ 5.44) in **42**.

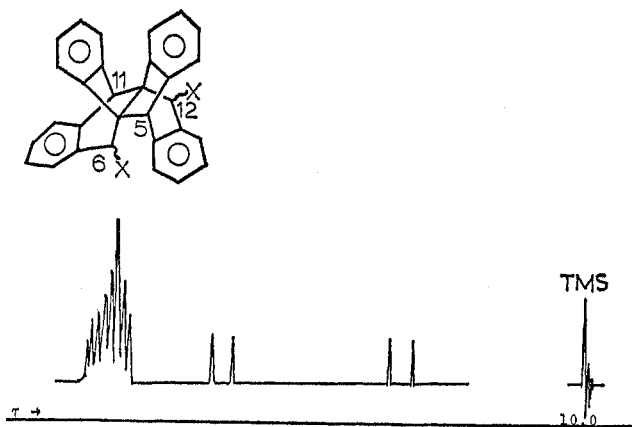


Figure 4.—Structure of a disubstituted *cis*-isojanusene and typical pmr spectrum. Omitted from spectrum are absorptions from protons contained in X.

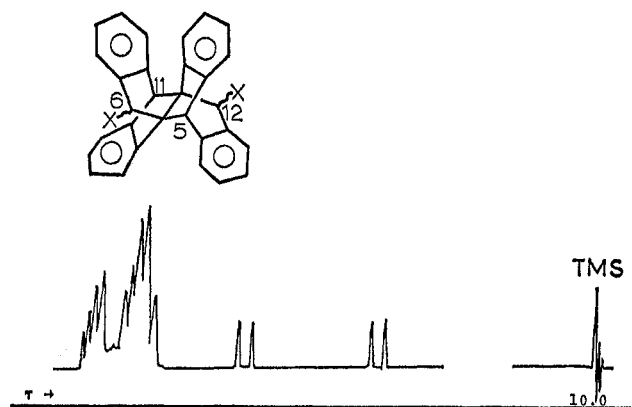


Figure 5.—Structure of *trans*-isojanusene and a typical pmr spectrum. Omitted from spectrum are absorptions from protons contained in X.

Isojanusenes.—Depending upon the configuration of the substituents, the pmr spectra of disubstituted isojanusenes **3** and **4** varies from two to four singlets, not including substituent and aromatic absorptions. *cis*-Isojanusenes and *trans*-isojanusenes have very similar spectra except for the aromatic proton absorptions, and their structures were later differentiated by X-ray crystallography.⁴ As indicated from Figures 4 and 5 and Tables IV and V, the pmr spectra show two sets of two aliphatic protons. Two protons have chemical shifts which are characteristic of hydrogens α to a given substituent, and the other two absorb at chemical shifts typical of benzydrylic protons. The general characteristic of having two groups of two hydrogens is consistent with the proposed structures (Figures 4 and 5).

As in the hemiisojanusene case, diacetate **36** has an acetate methyl absorption at an unusually high chemical shift (τ 8.80) and one at τ 7.89. Examination of Fieser models clearly indicates that exo (quasi-axial) substituents are positioned in the face of a neighboring benzene ring and therefore should be strongly shielded. These same models also indicate that the exo position ought to be sterically hindered. Because the hydrogens at C-6 and C-12 are not equivalent in the pmr spectrum

of **36** and because of the observations just noted, diacetate **36** is assigned the exo,endo configuration.

Diacetate **37** is assigned the endo,endo configuration, because both acetate methyl absorptions are at τ 8.00, which indicates that the substituents are endo and equivalent. This structure is preferred to the exo,exo configuration because the chemical shift resembles the endo acetate methyl absorption more than the exo. This compound also was prepared by treatment of **36** under thermodynamic conditions,² and molecular models had suggested that the "diendo" compound should be more stable than the "diexo" isomer.

The configuration of the substituents in the *trans*-isojanusene system is known with less certainty than in the *cis*-isojanusenes, because functional groups in *trans*-isojanusenes are not subject to strong shielding effects like those observed in the latter. Diacetate **42** is assigned the "diendo" configuration, since it was prepared under thermodynamic control² and Fieser models indicate that the endo,endo epimer should be the most stable. Also, the pmr spectrum shows that the hydrogens at C-6 and C-12, which have the same chemical shift, are equivalent. The exo,endo isomer was detected in the product mixtures in low concentrations. Since the two protons at C-6 and C-12 have different

chemical shifts, we assume that they have different configurations. The chemical shift of the proton at C-12 in **41** was about the same as that in **42**, and therefore we assign it the same configuration. That is to say, the hydrogen is assigned exo and the acetate substituent is designated endo.

Registry No.—5, 14707-22-3; 6, 23646-38-0; 7, 14596-96-4; 8, 29179-06-4; 9, 29179-07-5; 10, 29309-29-3; 11, 29309-28-2; 12, 23646-39-1; 13, 29308-17-6; 14, 29309-34-0; 15, 29428-03-3; 16, 29309-30-6; 17, 29309-31-7; 18, 29308-18-7; 19, 29308-19-8; 20, 29246-50-2; 21, 29308-21-2; 22, 29308-22-3; 23,

29428-06-6; 24, 29308-23-4; 25, 29309-37-3; 26, 29308-24-5; 27, 29308-25-6; 28, 29428-08-8; 29, 29308-26-7; 30, 29246-49-9; 31, 29246-46-6; 32, 29246-48-8; 33, 29308-30-3; 34, 29308-31-4; 36, 29246-47-7; 37, 29308-33-6; 38, 29179-08-6; 39, 29308-35-8; 40, 29339-43-3; 41, 29309-25-9; 42, 29309-26-0; 44, 29309-27-1; 45, 29595-83-3.

Acknowledgments.—The authors are indebted to the National Science Foundation and to the Institute of General Medical Sciences (Public Health Service Grant GM-12139) for support of this work.

Bridged Polycyclic Compounds. LXIX.

Preparation and Structures of the Diketoisojanusenes¹

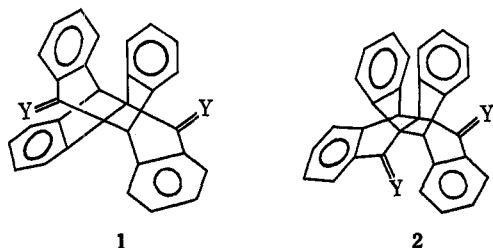
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Received August 6, 1970

Oxidation of the 6,12-diols of *trans*- and *cis*-isojanusenes led to the isomeric 6,12-diketojanusenes (**1b** and **2b**). The *trans* diketone **1b** is achiral and X-ray crystallographic data permit the lower melting isomer to be assigned that structure.

In the course of our work^{1,2} on the stereochemistry of the rearrangement reactions of derivatives of janusene (5,5a,6,11,11a,12-hexahydro-5,12:6,11-di-*o*-benzenonaphthacene),^{3,4} it became necessary to distinguish *trans*-isojanusene (**1a**) and its derivatives from *cis*-



a, Y = H₂
b, Y = O

isojanusene (**2a**) and its derivatives.⁵ To this end we prepared 6,12-diketo-*trans*-isojanusene (**1b**) and 6,12-diketo-*cis*-isojanusene (**2b**) from oxidation of the corresponding alcohols.^{2b} Although the pmr spectra and infrared spectra of **1b** and **2b** differed, they could not be used to distinguish between structures **1** and **2**. The lower melting diketone (mp 334–335°) gave a pmr spectrum in chloroform-*d*₁ with a singlet at τ 4.97 and aromatic proton absorptions at τ 1.94, 2.06, 2.70 and 3.07. The higher melting diketone (mp >360°) gave a pmr spectrum in chloroform-*d*₁ with a singlet at τ 4.96 and aromatic proton absorptions at τ 2.25, 2.35, 2.72 and 2.99. The infrared spectra were very similar except that the high melting isomer gave strong absorp-

tions at 1248 and 904 cm⁻¹ which were absent in the low-melting isomer. The latter, however, gave a medium absorption at 997 cm⁻¹ which was not present in the high-melting diketone.

Examination of the structures indicated that **1** should be achiral (meso), while **2** should be chiral. Because of the small amounts of compounds **1b** and **2b** on hand, and the difficulties involved in their preparation, we employed X-ray crystallography in order to distinguish between them rather than the usual chemical resolution techniques. We hoped that simple determination of the space group and of the number of molecules per unit cell might be sufficient to assign unequivocally a meso structure to one of the diketones. Fortunately this hope was realized and it has thus been possible to identify the meso compounds on the basis of simple symmetry arguments.

Neither of the two sets of crystals gave a good diffraction pattern. Only the lower melting ones gave a pattern that was adequate for space group determination, and, in this case, no reflections were observed at a Bragg angle greater than about 40° (Cu K α radiation). All the crystallographic work was carried out on the lower melting isomer. The crystallographic data obtained follow: system, monoclinic; $a = 9.09$ Å, $b = 8.62$ Å, $c = 14.74$ Å, $\beta = 112^\circ 15'$; systematic absences, (0*k*0) with *k* odd; number of molecules/unit cell, 2 (assuming a crystal density of 1.28 g/cc). These data were consistent with either of the space groups $P2_1$ or $P2_1/m$.

This isomer was then identified as the meso isomer since the *dl* isomer can be excluded from either space group as follows. Let us suppose that the isomer is, in fact, the *dl* isomer; then a unit cell containing two molecules must have one *d* enantiomorph and one *l* enantiomorph. If the cell should have space group $P2_1$, these two enantiomorphs would be related by a twofold screw axis, or the molecule itself would have to contain a twofold screw axis. The former alternative is inadmissible

(1) Previous paper: LXVIII. S. J. Cristol and M. A. Imhoff, *J. Org. Chem.*, **36**, 1861 (1971).

(2) (a) S. J. Cristol and M. A. Imhoff, *ibid.*, **36**, 1849 (1971); (b) S. J. Cristol and M. A. Imhoff, *ibid.*, **36**, 1854 (1971).

(3) S. J. Cristol and D. C. Lewis, *J. Amer. Chem. Soc.*, **89**, 1476 (1967).

(4) S. J. Cristol and W. Y. Lim, *J. Org. Chem.*, **34**, 1 (1969).

(5) The trivial nomenclature used for these compounds has been described.^{2a}