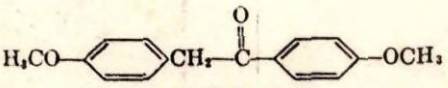
CRYSTALLOGRAPHIC DATA

No. 179. Deoxyanisoin

HARRY A. ROSE and JOHN G. WILLIAMS
Eli Lilly and Co., Indianapolis 6, Ind.



Structural Formula

DEOXYANISOIN can be crystallized from ethanol or benzene. In both cases the resulting crystals are needles elongated parallel to the *b* axis. The crystals usually show the 001 and $\bar{1}01$ faces and occasionally the 100.

The x-ray powder diffraction data were obtained using a camera 114.6 mm. in diameter and copper radiation with nickel filter. A wave length value of 1.5405 A. was used in the calculations. The indexing was done on the basis of a single crystal rotation pattern and a Weissenberg pattern around the *b* axis.

CRYSTAL MORPHOLOGY

Crystal System. Monoclinic.
Form and Habit. Needles elongated parallel to *b*.
Axial Ratio. $a:b:c = 3.905:1:2.815$.
Beta Angle. 135° 52′.

X-RAY DIFFRACTION DATA

Cell Dimensions. $a_0 = 21.71$ A., $b_0 = 5.56$ A., $c_0 = 15.65$ A.

X-Ray Powder Diffraction Data

d	I/I_1	*hkl*	*d* (Calcd.)	*d*	I/I_1	*hkl*	*d* (Calcd.)
11.04	0.13	001	10.90	2.92	0.01	212	2.91
7.93	0.13	$20\bar{2}$	7.83	2.73	0.05	004	2.73
5.50	0.05	002, $40\bar{2}$	5.45, 5.43	2.59b	0.08	220, 121	2.61, 2.59
5.21	0.05	110	5.22	2.45	0.03	022	2.45
4.91	0.08	011	4.95	2.41	0.03		
4.83	0.05	201	4.80	2.32	0.05		
4.48	1.00	210	4.48	2.29	0.05		
4.31	0.67	111	4.31	2.25	0.08		
3.86	0.13	012	3.89	2.21	0.08		
3.81	0.67	400	3.80	2.17	0.05		
3.41	0.26	202	3.41	2.07	0.08		
3.12	0.20	410	3.13	2.02	0.01		
3.08	0.40	$10\bar{5}$, $60\bar{5}$, 013	3.07, 3.06, 3.04				

Formula Weights per Cell. 4.
Formula Weight. 256.30.
Density. 1.287 grams per cc. (flotation), 1.296 grams per cc. (x-ray).

OPTICAL PROPERTIES

Refractive Indices. $\alpha = 1.524$, $\beta = 1.624$, $\gamma > 1.70$.
Optic Axial Angle. $2V = 85°$ (estimated).
Optic Axial Plane. Perpendicular to 010.
Optic Sign. Positive.
Extinction. $\alpha \wedge a = 8°$ in obtuse β.
Orientation. $\beta = b$.

FUSION BEHAVIOR. Deoxyanisoin melts in the range 110.0–11.5° C. as determined on a Kofler hot stage. No polymorphism was observed.

CRYSTALLOGRAPHIC data for publication in this section should be sent to W. C. McCrone, 501 East 32nd St., Chicago 16, Ill.

INFRARED QUANTITATIVE ANALYSIS DATA

Composition of Methyl Methacrylate–Butadiene Copolymers

R. M. B. SMALL, Polymer Corp., Ltd., Sarnia, Ontario, Canada CS-71

Component No.	Component Name	Component Formula	Range %	Accuracy %	λ or ν B. L. Pts.	Slit (mm) Δλ or Δν	Concn. w/w % Length cm
1	Polymethyl methacrylate	—$C_5H_8O_2$—	0–100	0.2 (duplicate)	1725 cm^{-1}	0.005 2.1 cm^{-1}	0.25 on MMA portion 0.05
2	*trans*-1,4-Polybutadiene	—C_4H_6—	5–100	0.6 on butadiene	967 cm^{-1}	0.0115 0.64 cm^{-1}	0.15 on butadiene 0.10
3	1,2-Polybutadiene	—C_4H_6—		0.6 on butadiene	909 cm^{-1}	0.0127 0.57 cm^{-1}	0.15 0.10

Instrument: Perkin-Elmer Model 21, NaCl prism
Sample Phase: Solution (1) dichloromethane; (2 and 3) carbon disulfide

Cell Windows: NaCl
Absorbance Measurement: Base line____ Point X
Calculation: Inverse matrix X Successive approx.____ Graphical____

Relative Absorbances[a]*–Analytical Matrix:* (concn. w/w %, length mm):

Component/λ	1725 cm^{-1}	967 cm^{-1}	909 cm^{-1}
1	4.31	0.317	0.124
2		2.99	0.035
3		0.264	3.87

Material Purity: Purified homopolymer, 99%; NBS *trans*-4-octene 99.8%; NBS 3-methyl-1-pentene, 99.5%.
Comments: MMA is industrial jargon for methyl methacrylate, both monomeric and polymeric.

[a] Relative absorbances are given as the slope of the Beer's law concentration curves used expressed in terms of absorbance per 100% of constituent.

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