cyanate and isocyanate, but it is not known which of these was produced.

Anal. Calcd. for $Ge(OCN)_4$: Ge, 30.17. Found: Ge, 30.35.

Germanium (iso)cyanate is a colorless liquid which rapidly hydrolyzes to germanium dioxide. A vapor pressure study, using the isotensiscopic method already described,[3] showed that thermal decomposition occurs slowly above 140°. The equilibrium vapor pressure values for the range 35 to 140° are expressed satisfactorily by the equation

$$\log_{10} p = 8.77 - \frac{2764}{T}$$

B. p. (extrapolated), 196°; molar heat of vaporization, 12.6 kcal.; m. p. −8°; density 24° (pycnometric), 1.7694; n^{25}_D 1.4793.

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[3] Laubengayer and Corey, *J. Phys. Chem.*, **30**, 1045 (1926).

DEPARTMENT OF CHEMISTRY
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ITHACA, N. Y.
A. W. LAUBENGAYER
LESLIE REGGEL

RECEIVED APRIL 17, 1943

Two New Thionacetophenetides

o-Thionacetophenetide was prepared in 26% yield by adding 18 g. of *o*-ethoxyphenyl isothiocyanate in ether to methylmagnesium iodide, from 32 g. of methyl iodide, 9 g. of magnesium turnings and 90 ml. of dry ether, refluxing for three hours, hydrolyzing and recrystallizing the resulting solid from 25% acetic acid, m. p. 70–71°. *Anal.* Calcd. for $C_{10}H_{13}NOS$: N, 7.17; S, 16.42. Found: N, 7.01, 7.14; S, 16.62, 17.34.

m-Thionacetophenetide was prepared in a similar manner and melted at 89–90°. *Anal.* Found: N, 6.90, 7.09; S, 17.26, 16.53.

We also prepared and analyzed *p*-thionacetophenetide and our melting point agrees with that of recent workers[1] rather than with that of Sachs and Loevy.[2]

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[1] Worrall, THIS JOURNAL, **46**, 2838 (1924); Kiprianov, Suitnik and Suich, *Chem. Abs.*, **30**, 4863 (1936); *J. Gen. Chem.* (U. S. S. R.), **6**, 42–9 (1936).

[2] Sachs and Loevy, *Ber.*, **37**, 876 (1904).

SCHOOL OF CHEMISTRY
RUTGERS UNIVERSITY
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LOUIS E. PERLGUT
D. L. COTTLE

RECEIVED JULY 12, 1943

COMMUNICATIONS TO THE EDITOR

LONG X-RAY DIFFRACTION SPACINGS OF THE KERATINS

Sir:

Recently, with techniques previously employed to determine the large fiber-axis period of collagen (640 Å.),[1] the following data were obtained for porcupine quill (α) and feather (β) keratins.

MERIDIONAL AND NEAR-MERIDIONAL REFLECTIONS

Porcupine quill tip			Feather rachis		
I	II	III	I	II	III
66	3	198	23.6	4	94.4
49	4	196	11.9	8	95.2
39	5	195	10.45	9	94.1
27.4	7	192	6.30	15	94.5
24.5	8	196	5.53	17	94.0
22.0	9	198	4.98	19	94.6
19.8	10	198	4.45	21	93.5
18.06	11	197.8	Layer-line reflections with *k* values of 2 and 4 to 13, inclusive, have been observed on the feather patterns		
15.2	13	198			
13.2	15	197			
12.36	16	197.8			
10.40	19	197.6			

I, Bragg spacing, in Å.; II, order no., *k*; III, *k* times spacing, in Å.

The low values listed in several instances result from considering meridional and near-meridional arcs together. This is unavoidable at present with porcupine quill patterns, since prominent large-spacing (83 Å.) layer-line components cannot be resolved satisfactorily, except on the equator, because of lateral diffuseness and lack of perfect orientation. The situation is better with feather, whose patterns show a strong, easily resolved row line indicating an important 34 Å. spacing transverse to the fiber axis.

In meridional directions on the patterns of both materials sharpness facilitates good resolution. The third order of porcupine quill and the fourth of feather are strongest and innermost in each case, and probably represent important structural features in the directions of the fiber axes.

The evidence clearly indicates fiber-axis periods of 198 and 95 Å., respectively, for porcupine quill and feather. On the eve of publication of these conclusions MacArthur[2] reported that periods of either 198 or 658 Å. are possible for porcupine quill, although the larger figure was favored and has been the only one anticipated previously.[3] It is impossible to deny categorically that periods

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[1] R. S. Bear, THIS JOURNAL, **64**, 727 (1942).

[2] I. MacArthur, *Nature*, **152**, 38 (1943).

[3] See W. T. Astbury, pp. 88–96 in "Advances in Enzymology," Vol. III, Interscience Publishers, Inc., New York, N. Y., 1943.

larger than those proposed herein may eventually be adopted, but no convincing reason for such a step is apparent now.

Fitting the porcupine quill data to 658 Å. is not critical, since no meridional spacings larger than one-tenth this value have been found, although they have been sought in this Laboratory. MacArthur's arguments were based largely on the position of a prominent short-spacing meridional arc (5.14 Å.), and on the expectation that a multiplier such as 128 $(2^{6}3^{0})$ should yield from it the true fiber-axis period. The 5.14 Å. arc is located in a region of considerable diffraction structure and may possess a layer-line component, so that its consideration is not an unambiguous matter. Also, a similar line of reasoning applied by Astbury to the collagen fiber-axis period was not conspicuously successful.[1,3]

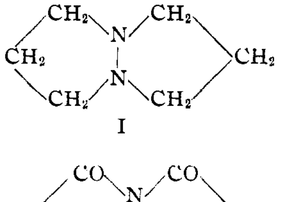
DEPARTMENT OF BIOLOGY AND BIOLOGICAL ENGINEERING
MASSACHUSETTS INSTITUTE OF TECHNOLOGY
CAMBRIDGE, MASSACHUSETTS RICHARD S. BEAR

RECEIVED AUGUST 12, 1943

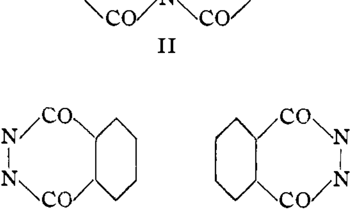
HETEROCYCLIC NITROGEN COMPOUNDS AND THE STEREOCHEMISTRY OF TERVALENT NITROGEN

Sir:

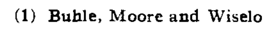
Buhle, Moore and Wiselogle[1] when reporting the preparation of 1,2-trimethylenepyrazolidine (I) claimed it as the first bicyclic compound of its type, and considered that in proposing to study the stereochemistry of this class of compound they would be approaching the problem of the resolution of tervalent nitrogen compounds in a new manner.



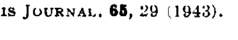
I



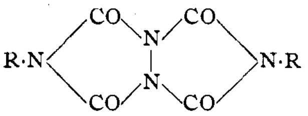
II



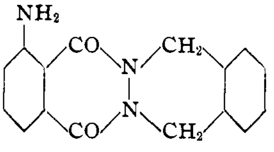
III



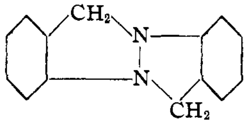
IV



(R = C_6H_5 or $C_6H_5{\cdot}CH{=}N{-}$)
V



VI



VII

Their first claim is open to dispute if they intend to claim that fused ring systems linked through vicinal nitrogen atoms are new. Two compounds of this class 2,3-phthalophthalaz-1,4-dione (III) and 3-phenyl-1,2-phthalopyrazoline (IV) were described by Drew and Hatt[2] and their preparation occasioned a search of the the literature for other compounds containing this structural feature. A number have been found, most of which are listed in the "Ring Index" of Patterson and Capell (Nos. 465, 535, 581, 608, 908, 1361, 2264 and 3463). Only two compounds (V) have been found of this kind which are not either specifically mentioned in the Ring Index or referred to in the literature it cites and they are of Ring Index Type R. I. 581.[3] Number 608 of the Ring Index (formula (II) above) is of interest since it contains the same condensed ring system as the trimethylenepyrazolidine (I) of Buhle, Moore and Wiselogle. Our concern here is to list the prior claims to have prepared compounds having this structural feature and not to consider the validity of those claims.

As regards their second claim for a novel approach to the problem of resolution of tervalent nitrogen compounds, it was appreciated when compound (III) was prepared that because of its structure it had especial interest with regard to the stereochemistry of tervalent nitrogen and this was mentioned.[2a] Substitution at the marked hydrogen atom of (III) renders the molecule, if non-planar, asymmetric and therefore theoretically resolvable. The preparation of such substituted compounds has been in progress for some-time and an account of the preliminary preparational work was recently submitted for publication to the *Journal of the Chemical Society.* In it the preparation of compound (VI) is de-

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(1) Buhle, Moore and Wiselogle, THIS JOURNAL, **65**, 29 (1943).

(2) Drew and Hatt, *J. Chem. Soc.*, 16 (1937); (a) *ibid.*, p. 19.

(3) Stollé, *Ber.*, **45**, 277 (1912).