

The *p*-nitrobenzoate and the 3,5-dinitrobenzoate were prepared from the alcohol and acyl chloride in pyridine and were much more easily obtained than the urethans. Some decomposition took place during the reaction but it was insignificant compared to that which took place in less basic solvents. The *p*-nitrobenzoate melted at 72.0–72.5° (*Anal.* Calcd. for C₁₀H₉NO₄: N, 6.77; C, 57.9; H, 4.38. Found: N, 6.80; C, 57.5; H, 4.59) and the 3,5-dinitrobenzoate melted at 108–109° (*Anal.* Calcd. for C₁₀H₈N₂O₆: N, 11.1; C, 47.6; H, 3.20. Found: N, 11.2; C, 47.4; H, 3.35).

The allophanate, prepared according to the method of Béhal,⁶ sublimed at 179–181° with decomposition. *Anal.* Calcd. for C₈H₈N₂O₈: N, 19.4; C, 41.6; H, 5.59. Found: N, 19.6; C, 41.3; H, 5.63.

(6) Béhal, *Compt. rend.*, **168**, 945 (1919).

SCHOOL OF CHEMISTRY
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NEW BRUNSWICK, NEW JERSEY RECEIVED JUNE 25, 1943

An Examination of the Fatty Oil from Buffalo Gourd Seed

By JOHN W. WOOD¹ AND HOWARD A. JONES²

The ground leaves, stems, fruit and root of the Buffalo gourd plant (*Cucurbita foetidissima*) are very attractive to several species of cucumber beetles.³ During an examination of the plant for its attractive principle, the physical and chemical characteristics of the oil from the seeds were determined. The plant is a member of the same genus to which the pumpkin belongs. Although pumpkin seed oil has been studied extensively, no examination of the oil from *Cucurbita foetidissima* seeds has been reported.

The seeds, resembling small pumpkin seeds, contain 29.7% of an oil extractable by ether. The crude ether-extracted oil has a yellowish-green color in thin layers and a brownish-red color in moderately thick layers. The oil has a bland, fatty taste, and a peculiar fatty odor. The physical and chemical constants of the crude oil are given in Table I. The percentages of saturated and unsaturated acids were determined by the lead-salt-ether method,⁴ and corrections were made for the small amount of unsaturated acids that are precipitated and weighed with the saturated acid fraction.

There is only a very small amount of glycerides of volatile acids present, as indicated by the low

(1) Present address: National Bureau of Standards, Washington, D. C.

(2) Present address: Bureau of Entomology and Plant Quarantine, Orlando, Fla.

(3) Elmore and Campbell, *J. Econ. Entomol.*, **29**, 830 (1936).

(4) Jamieson, "Vegetable Fats and Oils," 1932, p. 351.

Reichert–Meissl and Polenske numbers. The low acetyl value, 7.51, indicates a small amount of glycerides of hydroxylated acids. The percentage of insoluble acids, 92.4, has been corrected for unsaponifiable matter and does not represent insoluble acids + unsaponifiable matter (Hegner number), which is commonly reported.

In general the characteristics of the oil are similar to those of pumpkin seed oil as reported by Riebsomer and Nesty.⁵

TABLE I
PHYSICAL AND CHEMICAL CONSTANTS OF BUFFALO GOURD SEED OIL

Density (30°), g./cc.	0.9179
Refractive index (Abbe 30°)	1.4728
Iodine number (Hanus)	140.8
Saponification value	190.2
Soluble acids, %	0.2
Insoluble acids, %	92.4
Acid value	16.5
Unsaponifiable matter, %	1.74
Reichert–Meissl number	0.23
Polenske number	0.14
Iodine number of unsaturated acids	151.2
Unsaturated acids (basis of oil), cor., %	85.67
Saturated acids (basis of oil), cor., %	5.59
Acetyl value	7.51

(5) Riebsomer and Nesty, *THIS JOURNAL*, **56**, 1784 (1934).

BUREAU OF ENTOMOLOGY AND PLANT QUARANTINE
AGRICULTURAL RESEARCH ADMINISTRATION
U. S. DEPARTMENT OF AGRICULTURE
BELTSVILLE, MD. RECEIVED MAY 21, 1943

NEW COMPOUNDS

Germanium (Iso)cyanate

The method used by Forbes and Anderson¹ for preparing the (iso)cyanates of silicon, boron and phosphorus has been found to be satisfactory for the preparation of germanium (iso)cyanate.² 59.1 g. of germanium tetrachloride was dissolved in 200 cc. of benzene, and 167 g. (an equivalent quantity) of powdered silver (iso)cyanate was added in small portions through a reflux condenser with shaking. The reaction proceeded rapidly with the evolution of considerable heat. The mixture was heated on a water-bath for one hour, allowed to cool, and filtered, the residue being washed with benzene. The filtrate was fractionally distilled and a yield of 45.3 g. (68%) of germanium (iso)cyanate was obtained as a fraction boiling between 195 and 199°. This gave no test for chloride. It behaved like a pure compound and was evidently not a mixture of the

(1) Forbes and Anderson, *THIS JOURNAL*, **62**, 761 (1940).

(2) The term (iso)cyanate is used here to refer to a product which may be the cyanate, isocyanate, or a mixture of the two.

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

Anal. Calcd. for $\text{Ge}(\text{OCN})_4$: 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_D^{20} 1.4793.

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

DEPARTMENT OF CHEMISTRY
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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 $\text{C}_{10}\text{H}_{13}\text{NOS}$: 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 workers¹ rather than with that of Sachs and Loevy.²

(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
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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		
15.2	13	198	with k values of 2 and 4		
13.2	15	197	to 13, inclusive, have		
12.36	16	197.8	been observed on the		
10.40	19	197.6	feather patterns		

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-mer-

idional 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 MacArthur² 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

(2) I. MacArthur, *Nature*, **162**, 38 (1943).

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

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