

ing nicotine to nornicotine probably by trans-methylation.

*Nicotiana glauca* contains principally anabasine. When grafted to *N. tabacum* roots<sup>3</sup> or when hybridized with the latter species<sup>4</sup> the leaves have been reported to contain predominantly anabasine. Repetition of these experiments in this laboratory has disclosed that the supposed anabasine of the grafts and of the hybrids is actually a mixture of anabasine and nornicotine with usually more nornicotine than anabasine. The difficult separation of anabasine from nornicotine in mixtures of the two was accomplished by repeated fractional crystallizations of the picrates and of the methylated picrates after removal of nicotine by the method of Smith and Smith.<sup>4</sup> To show that the nornicotine present in the leaves of these plants actually arose *in situ* and at the expense of nicotine translocated from the roots, *N. glauca* scions were grafted to *N. tabacum* roots. After a period of growth, a tomato scion was grafted to the apex of each *N. glauca* scion. Ultimately, these three-tiered plants were examined for alkaloids. The *N. glauca* scions contained one part of nicotine to fifty parts of mixed anabasine and nornicotine, while the tomato scions contained only nicotine.

It is now clear that the replacement of the methyl group of nicotine in the plant leaf by the hydrogen atom of nornicotine accounts for the increase in secondary amine content (previously attributed to anabasine<sup>4</sup>) of such graft combinations and genetical hybrids. It follows that expectations<sup>4</sup> of the development of hybrids between *N. tabacum* and *N. glauca* that would be suitable for the commercial extraction of anabasine are without justification.

(3) Schmuck, Kostoff and Borozabina, *Compt. rend. acad. sci. U. R. S. S. (Doklady)*, **25**, 477 (1939).

(4) Smith and Smith, *J. Agr. Research*, **65**, 347 (1942).

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#### RESOLUTION OF 9-HYDROXYFLUORENE-2-CARBOXYLIC ACID

Sir:

The question whether fluorene has a folded or planar structure is a subject of controversy.<sup>1</sup> It might be thought that the classical methods of stereochemistry should furnish an answer but their application has been precluded by the inability to resolve fluorene compounds containing a 9-asymmetric carbon atom.<sup>2</sup>

We have now succeeded in resolving 9-hydroxyfluorene-2-carboxylic acid into its dextro and levo isomers.

(1) Rieveschl and Ray, *Chem. Rev.*, **23**, 378 (1938).

(2) Bennett and Noyes, *Rec. trav. chim.*, **48**, 895 (1929); *This Journal*, **52**, 3437 (1930); Badler, Thesis, Ludwig Maximilian University of Munich, pub. Vienna, 1926.

To 11.3 g. of 9-hydroxyfluorene-2-carboxylic acid (m. p. 240°) in 125 cc. of 95% ethanol was added 16.7 g. of strychnine in 125 cc. of chloroform. The resulting solution was distilled to remove the greater part of the chloroform. It was then filtered and placed in a pan of hot water and the whole allowed to come to room temperature. The following morning crystals in the form of rosetts, weighing 13.2 g., were obtained. These, recrystallized from 700 cc. of ethanol, sintered at 190° and melted at 203°. This strychnine salt was dissolved in a mixture of 100 cc. of ethanol and 200 cc. of 1% sodium hydroxide. This was poured into a liter of water containing 20 cc. of 6 *N* hydrochloric acid. The white fluffy precipitate was recrystallized from 60 cc. of ethanol and fine needles melting at 263° (block) were obtained.

*Anal.* Calcd. for C<sub>14</sub>H<sub>10</sub>O<sub>3</sub>: mol. wt., 226. Found: mol. wt. (pinene dibromide), 237; equivalent weight, 226.

These had the following rotations for red, yellow and green light for a solution of 0.7000 g. in 50 cc. of ethanol in a 2-dm. tube:  $\alpha^{27}_{656.3} + 0.82^{\circ}$ ;  $\alpha^{27}_{589.3} + 1.11^{\circ}$ ;  $\alpha^{27}_{516.3} + 1.39^{\circ}$ ;  $[\alpha]^{27}_{656.3} + 21.3^{\circ}$ ;  $[\alpha]^{27}_{589.3} + 39.6^{\circ}$ ;  $[\alpha]^{27}_{516.3} + 49.6^{\circ}$ .

The levo isomer was isolated similarly. It melted at 260° (block). A solution of 0.7500 g. in 50 cc. of ethanol in a 2-dm. tube gave:  $\alpha^{27}_{656.3} - 0.83^{\circ}$ ;  $\alpha^{27}_{589.3} - 1.09^{\circ}$ ;  $\alpha^{27}_{516.3} - 1.35^{\circ}$ ;  $[\alpha]^{27}_{656.3} - 27.6^{\circ}$ ;  $[\alpha]^{27}_{589.3} - 36.3^{\circ}$ ;  $[\alpha]^{27}_{516.3} - 45.0^{\circ}$ .

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#### INTRODUCTION OF THE ANGULAR METHYL GROUP

Sir:

In a previous report<sup>1</sup> we described the preparation of *cis*- and *trans*-9-methyldecalone-1, using Koebner and Robinson's excellent method for directing the alkylation to the angular position,<sup>2</sup> complemented by a scheme for removal of the protective arylidene group. Although this affords a good source of these particular ketones, there are, as recently pointed out by Birch and Robinson,<sup>3</sup> certain objections to our method from the point of view of more general applicability. We have been engaged for some time in a search for another protective group more easily removed than the arylidene group, and are reporting some of our findings now, because of the appearance of work along somewhat similar lines recently announced by the English workers.<sup>3</sup>

The condensation of decalone-1 with ethyl formate according to a previously described procedure<sup>1</sup> gave excellent yields of the colorless

(1) Johnson, *This Journal*, **65**, 1317 (1943).

(2) Koebner and Robinson, *J. Chem. Soc.*, 506 (1941).

(3) Birch and Robinson, *ibid.*, 591 (1944).

(4) See the preparation of 2-formyl-3-methylhydrofluorene-1 (Johnson, Anderson and Shih-Dang, *This Journal*, **66**, 198 (1944)).