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UNIVERSITY OF CALIFORNIA

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DEPARTMENT OF CHEMISTRY
LOS ANGELES, CALIFORNIA

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Dr. Melvin Calvin
University of California
Berkeley, Calif,

Dear Calvin,

I had a long talk with Wilmarth the other day and as a consequence of that, and your letter, there are a number of things tp tell you and a number of questions to ask you. First, as to the the information I want. As you probably know, I am going to do some more work on the deterioration problem and in connection with that I have decided to run capacities on the samples I work with. I have run a couple and got fair checks with what was reported, but there was some question in my mind as to what is meant by capacity. Is it the percentage of oxygen in the oxygenated sample, or the gain in weight of the deoxygenated sample? I suppose this is defined in some old report but I'm lazy.

I think there is some promise in my attempt to reactivate a deteriorated sample, but the measurement is still incomplete. I think I have got a 0.6% sample up to around 3% but this is to be checked. Will you please describe for me your procedure for measuring capacity: temperature of deoxygenation, and time, conditions of cooling and weighing, time of reoxygenation, etc. It seems to me that unless two persons used pretty nearly the same technic the results might be different to the extent of a few tenths of a percent. For example, one of duPont's samples was reported to me to have a capacity of 1.0%. I found 1.7%, and the difference might lie in my deoxygenation temperature (170°), which may have reactivated somewhat.

I asked Wilmarth to find out for me whether you were satisfied as to the identity of the N-methyl triamine from duPont. We are having our troubles trying to prepare it in an unambiguous way and if you are satisfied with the stuff you have we'll go on to something more available. It was my feeling that they may have prepared it in the same way as prtn, only using methylamine instead of ammonia; and in that case I wouldn't place my trust in it.

We have some, and will have more, 6-methylsalicylaldehyde and we are preparing the intermediates for the series of 4-hydroxy-6-alkylsalicylaldehydes I mentioned to you in a recent letter. Our o-ethylphenol hasn't arrived but when it does I think we can promise you the 3-ethyl compound. We are getting set up to run lithium metalations and hope to get to

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Dr. M. Calvin

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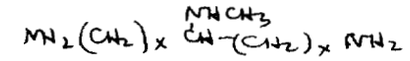
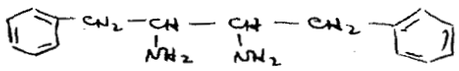
a number of 3- and 6-substituted compounds pretty directly. For example, we are sure we can make the 6-methoxy and probably we can get other 6-alkoxy compounds.

The triamine situation looks pretty dark at the moment. We had some great plans but the molecules haven't been behaving. We hope to get tris-aminomethylmethane and maybe a monoalkyl derivative but this is still at the stage of methane triacetic acid. The outlook for a compound of the type

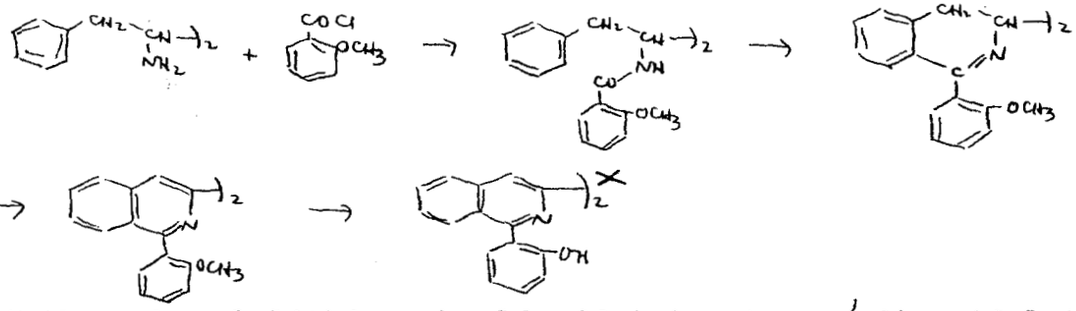


doesn't look very bright but we haven't given up hope yet.

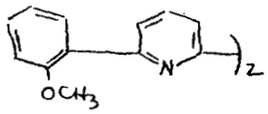
The hydroxyphenylpyridine series is very intriguing and I have a few ideas which, like most ideas on our compounds, depend on some tricky starting materials. I think if I could get



I could prepare the following compound by the Bishler-Napieralski isoquinoline synthesis, as follows:



o-Methoxyphenyl lithium should add twice to α,α'-dipyridyl to give



but the trouble again is to get the dipyridyl. It can be made but it is a mess; Eastman sells it at $4/gram. The monocompound should be easily attainable by adding o-methoxyphenyl lithium (or Grignard) to pyridine. But I'd like to make the bis-compound and I think the isoquinoline compound above should be the answer. It would be nice to put one or more $-CH_2-$ in the spot marked X on the bisisoquinoline above. This should be possible by the same series.

I was sorry to hear that you got neither vacation nor pay, and I can't think why since we had no trouble arranging it. If you get down, or I get up there, there are a lot of interesting things to report from Buffalo and a powwow with C.C.F. and Diehl.

I'll try to do some shipping soon.

Yours,

T. A. Geissman

T. A. Geissman

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