Still a slight impurity!!

Trace impurity	SL-2-139 a	acetone-d	Chemistry
trityl HG + STD	SL-2-139 b	$CDCl_3$	Chemistry
" "	SL-2-139 c	$CDCl_3$	Chemistry
" "	SL-2-139 d	$CDCl_3$	"

worked same as NMR Related BS — hopefully I can filter & remove solvent from my last HG rxn — I also need to make some stock solns for pzn's I will run next week

(~ 12pm — 5pm 8:30pm — 11pm)

6/22/03 Well — I filtered my HG — started freezing the soln so I got the pump for the solvent — Fucking piece of German shit glassware starts a major series of cracks — I barely was able to salvage the material — it was impossible to complete out —

They should change the name from Schott to Shit — This stuff is the worst glassware I've ever had to work with, period! — even the Chinese shit is better.

Anyway — I pumped the material down the best that I could & started recrystallizing it from CH_2Cl_2/hexane.

cleaned glassware as usual

(~2pm ~ 11pm)

6/23/03 Made up the following solns for a suspension pzn of IR (I'll target 60°C) — note that using these types of water systems (alcohols/H_2O & salts/H_2O) for such pzn's is my idea — No one had suggested using these earlier to me.

got they will try to take credit

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6/23/03

Methanol / H_2O soln:

~ 68g MeOH combined with 32g H_2O — according to CRC mp ~ −96.30°C

LiCl/NaCl/H_2O soln:

~23g LiCl, ~1.2g NaCl, ~75.8g H_2O combined mp ~ −77°C
According to Akopov et al.

Note – this is my idea to try suspension pzn of IB with HG — I believe it might work – if it does, emulsion pzn may will be even better most likely – but really suspension is the way to go since product is easy to separate

My plan will be to pzn 5.5mL IB (@ −78°C) in about 18mL hexane in say ~50mL of each aqueous stock soln — what I want to do is omit PT & cumylCl (protic initiation will of course be working) & use HG dissolved in C_7H_8 as before (essentially keep as many experimental parameters as unchanged as possible).

Took HG filtrate & concentrated this stuff.

(~ 9:40am - 5pm ~ 7:30pm – 11pm)

6/24/03

Ran NMR of mercury trimer SL-2-140-a & [illegible] [illegible] Chemistry
impurity

Spent a lot of of today helping Karen

Made a some stock solns —

HG stock in tol C_7H_8

took 0.4323g & dissolved in C_7H_8 to make 10mL soln thus for (5×10^{-5} moles)
0.04180g need to inject 0.969mL

Cumyl-Cl stock in hexane

took 0.103g & dissolved in C_6H_{14} to make 25mL soln thus for 5×10^{-6} mole (7.73×10^{-4}g) need to inject 0.188mL

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Since also using pt stock 1.0 ml need 11.388 g hexane & 5.5 ml IB

Thus — 11.388 g hex (~18.02 ml) & 1.0 ml trioctylAl were combined to which 5.5 ml IB was vac transferred & the whole was warmed to −78°C & stirred for ½ hour before vac transferring both solvent & monomer to the reactor @ which mixture was warmed & held @ −91.0°C ± 1°C via use of 50/50 dimethylether/heptane bath to which N_2(l) was added periodically. Next 1.0 ml PT stock (5×10^{-4} moles) then 0.970 ml HG stock (5×10^{-5} moles) then 0.190 ml cumyl-Cl stock were injected. After about 5 min pzn mixture was very viscous. Rxn was allowed to go for 1 hour before quenching with 1 ml MeOH/MeCN.

1.967 g or 50.83%

Again — 11.388 g hex (~18.02 ml) & 1.0 ml trioctylAl were combined to which 5.5 ml IB was vac transferred & the whole was stirred for 1 hour @ −78°C before vac transferring volatiles to the reactor² — reactor was held @ −91°C ± 1°C using same bath technique as before — Next 1.0 ml PT stock (5×10^{-4} moles) then 0.970 ml HG stock (5×10^{-5} moles) then 0.190 ml cumyl-Cl stock were injected. After 5 min the pzn mixture was thicker but not quite as thick as the previous exp — rxn was quenched after 1 hour with 1 ml MeOH/MeCN

1.872 or 48.37%

To ~37.5 ml MeOH/H_2O #1 (PG 140) stock was added 18.5 ml hexane & then vac transferred 5.5 ml IB. The mixture was subjected to 3 freeze/pump/thaw cycles & then warmed to −60°C* @ which 1.0 ml HG stock (5×10^{-5} moles) was injected. Rxn was stirred for 1 hour — product mixture poured into sep funnel — organic layer not viscous may be too much CT if any rxn occurred. Anyway — did not smell much IB gas nor did much gas evolve when I added additional H_2O & hexane to the sep funnel.

Temp was initially −64 rose to −60 then −58 then fell back to −64

* using dry ice CH_3CN

0.2955 g (after having blown off all volatiles!) or 7.64% yield

(~9:50 AM — 1 AM)

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