PROCEDURES OF PRODUCING
Methylphosphoryldifluoride
the precursor of SARIN

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Abstract : *Five steps of producing Methylphosphoryldifluoride are described with adequate details to show the procedures but not for producing because some data such as weights of raw materials and operating conditions are not disclosed. The author also suggested that SARIN may be useful in medicine such as for curing post-traumatic stress disorder, help people to forget bad experience or secret stories, by inhibiting or destroying synapses of specific nerves in certain part of the brain.*

What is Methylphosphoryldifluoride? The answer can be found from several websites on the internet. Some websites give incorrect information about the production process such as only four chemicals are required to produce it. Knowledge about SARIN can also be found in several websites including how to produce it using Methylphosphoryldifluoride and IPA (Isopropyl Alcohol).

1. Raw materials required to produce Methylphosphoryldifluoride.

1) PS2 = Decaline $C_{10}H_{18}$ 98%
2) PL2 = Methanol CH_3OH 96.5%
3) PS1 = 3,3-Dimethyl, 2-Butanol $CH_3C(CH_3)_2CHOHCH_3$ 98.5%
4) PH1 = Tributhylamine $N(CH_2CH_2CH_2CH_3)_3$ 98%
5) PH2 = Sodium Fluoride NaF (powder) 97.8-98.4%
6) PS3 = Sodium Sulfide Na_2S (solid) 60%
7) PS8 = Thionylchloride $SOCl_2$ 99.5%
8) PS4 = Phosphorustrichloride PCl_3 99.5%
9) PL3 = Chlorobenzene C_6H_5Cl 99%
10) PL4 = 2-Chloroethanol $Cl\text{-}CH_2CH_2OH$ 99%
11) PW = Process Water H_2O (Demineralizer 10 – 18 MegaOhm)

2. Codes of Chemicals, Mixers, and Reactors

Step No.	Codes of Chemicals	Code of Mixers	Code of Reactors
Step 1	Raw materials PS1, PS2, PL2, and PH1 Intermediate PM1	M-22 (SS 316L)	R-22 and R-23 (SS 316L)
Step 2	Raw material PH2 Intermediate PM2	None	R-62 (Glass Lined)
Step 3	Raw materials PS4, PS8 Intermediate PM3	M-27 (Glass Lined)	R-26 (Glass Lined)
Step 4	Raw materials PS3, PL3, PW Intermediate PM4	M-30 (Glass Lined)	R-30 (Glass Lined)
Step 5	Raw material PL4 Intermediates PM3, PM4	None	R-33 (Glass Lined)

Notes: 1) There are various vessels to receive distillate, intermediate and wastewater too.
2) All mixers are equipped with agitator, temperature probe, pressure indicator, valves, nozzles, and a dosing pump at the bottom (except for M-27).

3) All reactors are equipped with agitator, packed column, temperature probe, pressure indicator, valves nozzles, and a drain valve.
4) An evaporator D-62 is needed in Step 1.The evaporator D-62 needs no agitator. New technology uses thin film evaporator in place of common evaporator. Still a common evaporator is required as back up.
5) The total volume of mixers and reactors for the production of 944 liters of Methylphosphoryldifluoride in industrial scale are as follows:
M-22 = 2500 lt., M-27 = 630 lt., M-30 = 630 lt., M-31 = 630 lt, R-22 = 20600 lt, R-23 = 12250 lt, R-62 = 855 lt., R- 26 = 2040 lt.,R-30 = 2040 lt., R-33 = 2040 lt.

3. STEP 5 The last step

The last step of producing Methylphosphoryldifluoride is to mix and boil three components together in R-33 at constant temperature while distillation. One is a raw material PL4, the others are intermediate chemicals produced in step 3 (PM3) and step 4 (PM4).

Reaction Summary PL4 + PM3 + PM4 = Methylphosphoryldifluoride.

Procedure

1. Pump X_{PL4} kilograms of pure PL4 from the storage tank to R-33.
2. Pump X_{PM3} kilograms of PM3 from the vessel V-28 to R-33.
3. Pump X_{PM4} kilograms of PM4 from the vessel V-32 to R-33.
4. Stir well and heat up R-33 until reflux occurs. Separate the distillate to a glass vessel V-65. Maintain the temperature of the liquid in R-33 by means of reflux and temperature control. Now we have Methylphosphoryldifluoride in V-65.
5. After finished the equipment is washed by PL4, drain and followed by caustic soda solution for cleaning.

4. STEP4 Procedure of producing PM4

Reaction Summary PM3 + PL3 + PS3 + PW = PM4 (after distillation)

Procedure

1. Pump $X1_{PL3}$ kilograms of pure PL3 into the reactor R-30.
2. Weight X_{PS3} kilograms of PS3 and fill it in R-30, then stir well.
3. Pour X_{PW} kilograms of PW (process water) into R-30, the mixture will be suspension. Heat up the reactor until reflux occurred and maintain.
4. Pump $X2_{PL3}$ kilograms of pure PL3 into the mixer M-30.
5. Pump X_{PM3} kilograms of PM3 from the vessel V-28 into the mixer M-30. Stir well.
6. Pump the mixture in M-30 slowly by dosing pump to R-30 while always keeping reflux continued through out the dosing.
7. After dosing finished, boil the mixture in R-30 at reflux temperature until the reactions stop. Measure and record the weight ratio of PL3 : PM4 obtained by titration.
8. Distill the mixture at head temperature and keep the distillate in V-31.
9. The bottom of R30 is poured into M-31, add Caustic soda solution, drain the suspension from R-30 into M-31. Stir well. Cool it down.
10. Wash R-30 with pure PL3. Stir well and drain the waste into M-31.

11. Stir the suspension in M-31 until all the solids are dissolved, the pH must be high enough. Stop stirring and let precipitation occur.
12. Two choices are to be chose. First choice is to distill PL3 in the waste and return to storage tank for the next batch. It requires another reactor to do this job. This is done in large production scale ,i.e. about 1,000 kilograms product per batch.. Second choice is dumping the waste to wastewater treatment plant. This is suitable for laboratory scale ,i.e. about 10 kilogram of product per batch.
13. The distillate in V-31 is still not pure enough. It is the mixture of solvent and product. Pump the distillate in V-31 into R-30 and distill PM4 at constant reflux ratio. The pure PM4 obtained is filled in V-32. Distillation stop when the head temp is reached.
14. Again we may distill the remaining PL3 in R-30 for the next batch or dump it away.

5. <u>STEP3</u> Procedure of producing PM3

Reaction Summary PM2 + PS4 + PS8 = PM3 (after distillation)

Procedure

1. Pump X_{PS8} kilograms of pure PS8 into R-26.
2. Pump X_{PM2} kilograms of PM2 from V-62 to mixer M-27 then add X_{PS4} kilograms of PS4 into M-27 through the nozzle and stir well.
3. Heat up R-26 and dose the mixture in M-27 by gravity to R-26 by controlling the flow in order to regulate the temperature of R-26 and the waste gas must not too much.
4. After dosing, stir slowly. Temperature rises up, then stir a while and stop.
5. The excess of PS8 in R-26 shall be distilled at small vacuum pressure and T_1degree Celcius. The distillate PS8 is kept in V-26 for using in the next batch.
6. Then, reduce pressure in R-26 to a vacuum of P1 Milli Bar and distill for the product at T2 degree Celcius , the distilled product PM3 is filled in V-28. Cool down R-26 and drain the waste into a plastic drum for further treatment.

6. <u>STEP2</u> Procedure of producing PM2

Reaction Summary PM1 + PH2 = PM2 (after distillation)

Procedure

1. Pump X_{PM1} kilograms of PM1 from V-60 to R-62 and heat up to distill excessive PS2 away to a plastic drum. Then, reduce the temperature of R-62 and purge with nitrogen twice.
2. Pour X_{PH2} kilograms of PH2 through the manhole of R-62. Stir well. Then, heat up to reflux temperature and keep the boiling temperature for a while. Then, heat up to reflux temperature and keep the reflux continue at all time. The temperature rises up keep on stirring then cool it down.
3. Reduce vacuum to P2 Milli Bar and distill the mixture at T_2 degree Celcius. The pure distilled PM2 will be kept in V-62 while the bottom of R-62 is dumped into a plastic drum for further treatment and disposal.

4. Wash R-62 with water and drain it to wastewater treatment plant, then add caustic soda solution into R-62. Heat up and distill until reflux occur, then leave the reactor cool down. Drain the wastewater into wastewater treatment plant and leave R-62 dry by evacuation.

7. <u>STEP1</u> Procedure of producing PM1

Reaction Summary PS1 + PL2 + PS2 + PH1 = PM1 (after distillation)

Procedure

1. Pump X_{PS1} kilograms of pure PS1 into V-22
2. Pump $X1_{PL2}$ kilograms of PL2 into mixer M-22 and pump X_{PS1} kilogram of PS1from V-22 into M-22. Stir well.
3. Pump X_{PH1} kilograms of PH1, pump $X2_{PL2}$ kilograms of PL2 and pump X_{PS2} kilograms of PS2 into R-22. Stir well and reduce temperature of R-22 not to have reflux.
4. Pump the mixture in M-22 by dosing pump intoR-22 slowly. Control temperature (the heat generated from the reaction can be controlled by the rate of dosing pump) After half of the mixture in M-22 is used there will be slurry of salt increasing in R-22. The temperature in R-22 will rise up when the mixture in M-22 is nearly exhausted. The reaction generates waste gas which must be scrubbed out. Stir R-22 well.
5. After finished dosing, the temperature in R-22 still rises up gradually. Stir well and Heat up R-22 slowly but lower than reflux temperature. Until the temperature is stable. Then stop stirring and allow precipitation to occur in R-22. As R-22 is too big to do further processing, so we need R-23 to do the job. Pump the clear top layer of R-22 into V-23 and pump the bottom sludge of R-22 into R-23. (The pump and pipe from R-22 to R-23 need heat tracing in order to prevent clogging.)
6. Heat up R-23 to distillate product to V-23. Now, we have not got PM1 in V-23 yet. The mixture in V-23 still required further fine evaporation with thin film evaporator or common evaporator.
7. The remaining liquid in R-23 shall be mixed with caustic soda solution and heat up gently. The liquid will be separated into 2 layers. The bottom part shall be drained to wastewater treatment plant, leaving the top layer which shall be mixed with process water. Again we may distill the remaining PH1 in R-23 azeotropically for using in the next batch or dump it away, depending on how big is the scale of production.
8. Pump the mixture from V-23 gradually to thin film evaporator or common evaporator for evaporation at P3 Milli Bar and below T3 degree Celcius. The bottom product is the required PM1 to be filled in V-60. The vapor is PL2 which can be condensed and pumped in to the storage tank of PL2 for the next batch.

Remarks: Methylphosphoryldifluoride + Isopropyl Alcohol (IPA) CH3CHOHCH3 99.7%
= SARIN.

Procedure of producing SARIN: By slowly gravitational dosage of IPA into Methylphosphoryldifluoride in R-33 while stirring in such a way that there is reflux at all time without heating, the ratio of Methylphosphoryldifluoride and IPA by volume is 5.64 : 10,waste gases are delivered to waste gas scrubber. After finished dosage of IPA then cool down the reactor to T1 degree Celcius and reduce the pressure of the reactor to P Milli bar to distill out waste gases for a while and heat up the reactor slowly to T2 to distill out SARIN as the product.

Applications Hint: SARIN may be useful in curing post-traumatic stress disorder by inhibiting or destroying synapses of specific nerves in certain part of the brain which control the memory which causes the syndrome. In addition, if we know which part of the brain controls which specific memory, sad experience or secret stories, then SARIN can be used to erase those sad experience or secret stories from the brain.

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