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MEMORANDUM

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HSM-05002

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DATE: March 10, 2005

SUBJECT: REVIEW OF CHLOROPICRIN EXPOSURE MONITORING STUDY, DATA PACKAGE 209842

I reviewed worker exposure monitoring data from Data Package 209842, which was submitted by the Chloropicrin Manufacturers Task Force (CMTF). This data package contains a two-volume study report titled, "Monitoring of Chloropicrin Emissions from Field and Greenhouse Drip Irrigation Applications, and Implied Worker Inhalation Exposure from Applications of Chloropicrin by Shank Injection, Drip Irrigation Systems and at Tree Replant Sites." This study (Rotondaro, 2004) was submitted by the CMTF in response to a reevaluation notice sent by Department of Pesticide Regulation (DPR) to chloropicrin registrants.

Worker exposure monitoring data consisted of air sampling tubes containing XAD-4 connected to air pumps calibrated at 50 ml per minute. One or two air samplers were attached to each worker collar; additionally, workers injecting chloropicrin with handwands for tree replanting had an air sampler attached to the lower leg to allow estimation of dermal exposure to chloropicrin (concentrations near the leg were anticipated to be higher during this application method).

This study is acceptable for use in DPR's Exposure Assessment Document (EAD) for chloropicrin. It conforms to the protocol accepted by DPR, and the quality assurance was acceptable. Results were reported in a way that differs from DPR practice: CMTF reported geometric means, and DPR practice is to use the arithmetic mean rather than the geometric mean as a central tendency estimate, with logarithmic distributions used to calculate upper bound estimates for short-term exposure (Powell, 2003). Additionally, CMTF provided 8-hour time-weighted average (TWA) concentration estimates that assumed two hours of non-exposure plus six hours exposure (i.e., reported concentrations had been multiplied by 0.75), while DPR estimates normalize workday exposures by assuming that workers are exposed for a full 8 hours. However, CMTF also reported "actual" concentrations that corresponded to the monitoring periods, and spreadsheets with the calculations were in Appendix D of Rotondaro (2004). Results reported in this memo used these "actual" calculations, rather than the CMTF-calculated 8-hour TWA concentrations.

One question that is not clear from the report is whether handler "exposure periods" included

times away from the field. Handler exposure times are longer than the reported application time, and the description of the application at some sites gives the impression that handlers might have been monitored before beginning activities that could result in exposure to chloropicrin. For example, the description of shank broadcast non-tarped shallow applications starts as follows (p. 26): "The driver arrived at each site with a truck. The driver unloaded the tractor and moved the tractor near the truck." Was the driver monitored during this time? Chloropicrin cylinders were not handled until after the truck and tractor were in position. Tasks covered by handler monitoring should be clarified. This question and others are discussed at the end of this memorandum.

Was the Study Conducted According to Protocol Submitted to DPR?

The protocol for this study was reviewed by DPR (Jones, 2002) and modified and amended at DPR's request (Cortez, 2002; Frank, 2003). The scenarios to be covered by this and previous CMTF studies were summarized by Powell (2002). All scenarios marked by Powell (2002) as requirements to be met are included in this study. A total of nine application methods were monitored: three shank broadcast methods (tarped shallow, non-tarped shallow, and non-tarped deep); two shank bedded methods (tarped shallow and non-tarped shallow); one surface drip method (tarped); one buried drip method (non-tarped); one greenhouse drip method (tarped); and one handwand method for tree replanting. For each of the nine methods, three separate sites were used, generally representing different California regions (i.e., desert, central or south coast, Central Valley). DPR recommended that desert monitoring occur during the high-use months of December through February (Cortez, 2002), and that was done (with the exception of the greenhouse application, Site 24).

Handler and reentry replicates were spread out among the three sites to give an acceptable number of total replicates. DPR specified a minimum of five replicates per scenario, and noted that "DPR does not consider multiple samples from the same individual performing the same task at the same site as replicates" (Jones, 2002). CMTF stated in the protocol that the replicates from this study, combined with those from Beard *et al.* (1996), will total five replicates per scenario. Table 1 summarizes handler replicates provided by each study.

There are some existing and proposed product labels with application rates greater than those monitored in the CMTF study. DPR agreed to application rates that were lower than maximum rates allowed on some products labels (Jones, 2002), stating that "DPR will adjust the exposure estimates proportionately using the maximum product label rate."

Table 1. Handler Scenarios Monitored by the Chloropicrin Manufacturers Task Force

Application Method	Study Reported in 2004 [a] Rate [c]	Study Reported in 2004 [a] Scenarios [d]	Study Reported in 1996 [b] Rate [c]	Study Reported in 1996 [b] Scenarios [d]	Total [e] per Scenario
Shank broadcast					
Tarped shallow (9-12" deep)	350	None	350	Tractor Driver (1 – AZ, 3 – WA, 6 – FL); Co-pilot (1 – AZ, 3 – WA, 6 – FL); Shoveler (2 – AZ, 6 – WA, 6 – FL)	10 - 14
Non-tarped shallow (4-13" deep)	175	Tractor Driver (4), Soil Sealer (4)	175	Tractor Driver (1), Soil Sealer (1)	5
Non-tarped deep (18-24" deep)	175	None		None	0 [f]
Shank bedded					
Tarped shallow (6-12" deep)	350	Tractor Driver (4), Co-pilot (3), Shoveler (3)	350 [g]	Tractor Driver (2), Co-pilot (1), Shoveler (2)	4 - 6
Non-tarped shallow (9-12" deep)	175	Tractor Driver (3)		Tractor Driver (1)	4
Drip					
Surface, tarped (1-3" deep)	300	Applicator (6)		None	6
Buried, non-tarped (10" deep)	300	Applicator (6)		None	6
Greenhouse, tarped (1-2" deep)	300	None		None	0 [h]
Handwand					
Replant (Probe to ~18" deep)	500	Applicator (6)		None	6

[a] Data from Table 1 in Rotondaro (2004). Monitoring done in several locations in California.
[b] Data from Tables 23 – 29 and pages 53 – 55 in Beard *et al.* (1996). Monitoring done in Phoenix, AZ; Yakima, WA; and Bradenton, FL (Jones (2002) stated DPR would pool these data).
[c] Target rate (lbs AI/acre).
[d] Handler activities monitored with application method (scenario is a combination of application method and activity). The number of replicates monitored for each scenario is listed in parentheses. Each replicate was defined here as a single worker at a distinct site (Beard *et al*. (1996) monitored the same worker more than once, but each worker is counted just once here).
[e] Combined number of replicates monitored in both studies. DPR target is five replicates per scenario (Jones, 2002); Powell (2002) suggested that four replicates would be acceptable.
[f] DPR waived the requirement for these handler replicates, and will rely on data from non-tarped shallow applications (Jones, 2002); only reentry exposures were monitored for this method (see Table 2).
[g] Rate was set at 350 lbs AI/acre; because beds were 50% of treated field, actual rate was 175lbs AI/acre (Beard *et al.*, 1996). The actual rate in Rotondaro (2004) is unclear, as rate was simply reported without reference to whether bedded area was considered.
[h] DPR waived the requirement for these handler replicates (Jones, 2002), provided that applications are made from outside the greenhouse, and that self-contained breathing apparatus is required for anyone entering a greenhouse before aeration is completed.

DPR also requested that replant site (treehole) handwand applications include air monitoring "near the applicator's lower extremities," due to DPR's concern about chloropicrin dermal exposure in that area because the injection wand is close to the applicator's feet (Jones, 2002). At the same time, DPR requested laboratory data on skin permeability to chloropicrin vapor, but stated that "if CMTF does not wish to provide this data, the octanol-water partition coefficient, with an uncertainty factor, will be used to estimate skin permeability." CMTF did the requested lower-extremity air monitoring (Rotondaro, 2004). Skin permeability data for chloropicrin were not provided by CMTF, nor are such data presently available in the literature.

Potting soil fumigation was mentioned in correspondence while the protocol was in review at DPR (Wilhelm, 2001; Cortez, 2002; Jones, 2002), but this use was not listed in the protocol. CMTF indicated an intention to support potting soil fumigation, and intended to have this use supported by data generated during handwand applications to tree replant sites (Wilhelm, 2001). DPR believes that the application methods are quite different, and that handwand application data cannot address exposures during potting soil fumigation (Jones, 2002). Cortez (2002) noted that potting soil was not addressed in the draft protocol, and asked if CMTF intends to support this use. I do not have a record of CMTF's response to this question; however, Powell (2002) noted that potting soil fumigation was "unsupported" by CMTF.

CMTF declined to generate data to support surface drip non-tarped application, or any gas application method (Wilhelm, 2001). CMTF indicated that these methods are not used with chloropicrin "as an active ingredient." DPR acknowledged that CMTF would not support these application methods (Jones, 2002).

Reentry scenarios that were monitored are summarized in Table 2. DPR requested that reentry workers be fitted with two pumps, one to sample for the first hour following reentry and one to sample for 4 hours. DPR requested 1-hour reentry samples because "DPR will consider a 1-hour duration when calculating peak exposure" (Jones, 2002). This monitoring was done, and results were reported in Appendix D of Rotondaro (2004).

Analytical Method

Appendix C contains the full analytical report (Maliani, 2004). Sorbent in the front and back sections of the air sampling tubes was extracted with ethyl acetate, and amount of chloropicrin in each sorbent section was determined using gas chromatography with an electron capture detector. The limit of quantitation (LOQ) was 0.1 µg chloropicrin per section (Maliani, 2004). DPR requested (Jones, 2002) that the LOQ for this study be the same as in the previous CMTF study. Although the LOQ of 0.1 µg/section (or 0.2 µg/tube, as each tube has two sections) reported by Maliani (2004) is greater than the LOQ of 0.07 µg/tube determined in the previous CMTF study (Beard *et al*., 1996), the difference is considered acceptable (0.2 µg/tube vs. 0.07 µg/tube is about a three-fold difference).

Quality Assurance

The analytical method was validated before use by fortifying three tubes each at 0.1 and 300 µg chloropicrin per tube (Maliani, 2004). Fortification was done by applying an appropriate amount of the fortification solution to the glasswool plug at the front of the tube, then drawing air through the tube. Recoveries of these fortified tubes ranged 84.0 – 96.0 %.

Table 2. Reentry Scenarios Monitored by the Chloropicrin Manufacturers Task Force [a]

Application Method	Days [b]	Reentry Scenarios Monitored [c]
Shank broadcast		
Tarped shallow	10 [d]	Soil Shaper (5); Tarp Splitter (15); Tarp Remover (29)
Non-tarped shallow	10	Soil Shaper (5)
Non-tarped deep	10	Soil Shaper (5)
Shank bedded		
Tarped shallow	7	Tarp Puncher (3); Tarp Splitter (1); Tarp Remover (3) [e]
Non-tarped shallow	7 [f]	Pipe Layer (6)
Drip		
Surface, tarped	10 [g]	Tarp Puncher (5)
Buried, non-tarped	NA	None
Greenhouse, tarped	NA	None
Handwand		
Replant	NA	None

[a] Data from Rotondaro (2004), unless otherwise indicated.
[b] Number of days until monitored workers entered to punch tarps, shape soil, or lay pipe. NA: Not applicable (reentry workers were not monitored following applications by this method).
[c] Activities monitored following application by specified method (scenario is a combination of application method and activity). The number of replicates monitored for each scenario is listed in parentheses. Each replicate was defined as a single worker at a distinct site.
[d] Tarps were split 5 days post-application and removed the next day; soil shapers were monitored 10 days post-application. Tarp splitter and tarp remover replicates for this scenario are from Beard *et al*. (1996), in which activities occurred 5 and 6 days post-application, respectively, in AZ; activities occurred 6 and 7 days post-application in WA and FL.
[e] The tarp splitter and tarp remover replicates for this scenario are from monitoring in AZ (Beard *et al*., 1996). Tarp splitting involves slicing a tarp lengthwise with a cutting disk, while tarp punching refers to punching a hole in the tarp through which planting may later be done. Rain occurred during reentry at Site 10.
[f] Reentry occurred 6 days postapplication at Site 14.
[g] Reentry occurred 5 days postapplication at Site 17.

Quality assurance samples consisted of control samples, procedural fortified tubes and field fortified tubes. Control tubes were set up at each site and air was drawn through them in the same way as the sample tubes attached to workers. Nearly all controls had chloropicrin < LOQ; the two exceptions contained chloropicrin amounts of 0.127 and 0.131 µg/section, just slightly above the LOQ of 0.1 µg/section. The controls are considered acceptable.

Field fortifications (FFs) consisted of triplicate XAD-4 tubes spiked with 2.0, 6.0, and 300 µg chloropicrin per tube. Tubes were fortified in the laboratory and stored on ice until monitoring began in the field, when they were attached to sample pumps that were running throughout exposure monitoring. FFs were done daily (except at Site 28, which shared FFs with Site 9). Mean FF recoveries are reported in Table 3. All but one site (Site 21) had mean FF recoveries within a range considered acceptable to DPR, 70 – 130% (ARB, 1999). At Site 21, all FF replicates of the mid-level (6 µg/tube) were elevated, possibly due to an error in fortification. However, all of the samples at this site ranged < 0.1 – 0.550 µg/tube, and would be adjusted based on the recovery of the low-level fortification (2 µg/tube). Thus, the unacceptable FF results do not influence the study, nor are they considered to be indicative of any systematic problem with the analysis.

Results

Breakthrough (chloropicrin residues recovered from back sorbent section as well as front section) occurred in several sorbent tubes, though in most (including all sample tubes) the amount in the back section was below the LOQ. Maliani (2004) presented tables summarizing breakthrough above the LOQ in procedural fortified (16 of 236 tubes) and field fortified (66 of 486) tubes. Except for one field fortified tube at the mid-level (6 µg chloropicrin per tube), all breakthrough above the LOQ occurred at the high level (300 µg chloropicrin per tube). This breakthrough was almost always less than 1%, and never exceeded 2.2%. These results are below the breakthrough criterion of 10% suggested by Huey and Materna (2002) for acceptable air sampling. Thus, breakthrough is considered to be at an acceptable level in this study.

Arithmetic mean concentrations based on data from Rotondaro (2004) for handlers and reentry workers are summarized in Table 4; these were the "actual" concentrations reported by Rotondaro (2004), rather than the adjusted (i.e., multiplied by 0.75) 8-hour TWA. Concentrations are reported in $\mu g/m^3$, but equivalent concentrations in parts per billion (ppb), expressed as ratio of weight of chloropicrin to volume of air, can be estimated using the ideal gas law. At 1 atmosphere of pressure and a temperature of 25°C, the concentration in ppb is equal to the concentration in $\mu g/m^3$, multiplied by 24.45 liter-atm/mole and divided by the molecular weight of 164.38 g/mole. As 24.45/164.38 = 0.1487, this value can be multiplied by the concentration in $\mu g/m^3$ to obtain the concentration in ppb.

The highest handler chloropicrin concentrations reported by Rotondaro (2004) were for drivers in the shank bedded non-tarped method (Sites 13 – 15); the arithmetic mean $\pm$ SD concentration was 255 $\pm$ 120 $\mu g/m^3$. Concentrations measured outside the cabs for these drivers were higher; the two replicates gave results of 208 $\mu g/m^3$ and 1,020 $\mu g/m^3$, for a mean of 614 $\mu g/m^3$. As mentioned above in Table 1, a single driver replicate was monitored in Beard *et al.* (1996). Including data from this replicate gives a mean $\pm$ SD of 207 $\pm$ 138 $\mu g/m^3$.

Table 3. Mean Field Fortification Recoveries During Air Monitoring for Chloropicrin [a]

	2 µg			6 µg			300 µg		
Site	**N**	**Mean**	**CV**	**N**	**Mean**	**CV**	**N**	**Mean**	**CV**
1	3	95.3	0.3	3	97.3	3.0	3	95.2	0.9
2	3	91.3	2.9	3	94.6	4.0	3	96.1	10.8
3	3	94.9	2.3	3	95.7	0.4	3	95.4	5.5
4	6	105	7.5	6	100	12.1	6	108	13.6
5	6	83.0	28.5 [c]	6	100	4.0	6	99.9	13.2
6	6	90.1	4.4	6	91.8	2.7	6	97.9	11.7
7	3	95.0	12.5	3	79.3	6.2	3	94.6	11.5
8	3	95.1	2.3	3	94.6	0.5	3	106	4.5
9 [b]	3	80.5	16.3	3	90.8	1.7	3	92.7	3.9
10	6	104	9.0	6	115	10.0	6	111	2.3
11	6	96.5	3.7	6	95.8	1.8	6	95.7	3.6
12	6	92.1	7.4	6	94.8	2.4	6	97.0	2.5
13	6	104	4.7	6	78.2	7.4	6	86.9	16.3
14	6	94.4	15.6	6	99.4	7.8	6	101	9.5
15	6	101	5.1	6	94.9	4.8	6	102	9.7
16	18	98.8	5.9	18	97.4	7.2	18	96.7	5.8
17	6	87.5	12.2	6	89.9	5.4	6	95.5	7.5
18	6	91.2	3.5	6	89.2	2.3	6	100	4.2
19	3	86.3	3.6	3	88.8	1.0	3	90.7	1.7
20	3	84.5	12.3	3	89.6	5.3	3	96.5	0.8
21	3	105	6.4	3	169 [d]	1.8	3	102	7.6
22	15	95.9	8.1	15	98.7	3.7	15	103	5.2
23	15	101	7.8	15	99.1	7.0	15	98.3	6.8
24	15	98.6	5.2	15	99.6	6.2	15	103	6.4
26	3	79.4	2.3	3	79.7	8.6	3	88.9	2.0
27	3	92.1	1.7	3	93.1	2.6	3	92.5	9.2

[a] Data from Table 11 in Rotondaro (2004). Arithmetic mean and coefficient of variation (CV); N = number of samples. Site 25 data omitted (site not used in study).
[b] Site 28 relied on field fortification data from Site 9.
[c] CV for the low-level fortification (2 µg/tube) at this site was high (> 25%), due to one replicate with 35.5% recovery. The other five replicates ranged 88.1 – 97.9.
[d] All replicates at the mid-level (6 µg/tube) were elevated at this site, possibly due to an error in fortification. Samples at this site ranged < 0.1 – 0.550 µg/tube, and would be adjusted based on the recovery of the low-level fortification (2 µg/tube). Thus, these results do not influence the study.

Table 4. Results of Worker Exposure Monitoring for Chloropicrin

	Handlers [a]		Reentry Workers [b]		Acres
Application Method	**Activity**	**Concentration [c]**	**Activity**	**Concentration [c]**	**Treated [d]**
Shank broadcast					
Tarped shallow	None	No monitoring	Soil Shaper	75.9 (79.5)	3.5-5.1
Non-tarped shallow	Driver	118 (60.9)	Soil Shaper	16.1 (7.69)	3.7-9.7
	Soil Sealer	66.6 (54.3)			
Non-tarped deep	None	No monitoring	Soil Shaper	11.0 (2.69)	2.7-5.1
Shank bedded					
Tarped shallow	Driver	121 (74.0)	Tarp Puncher	8.97 (3.62)	1.1-1.9
	Co-pilot	166 (128)			
	Shoveler	96.8 (61.8)			
Non-tarped shallow	Driver	255 (120)	Pipe Layer	8.84 (0.982)	1.7-5.6
	Outside cab	614 (574)			
Drip					
Surface, tarped	Applicator	69.3 (31.7)	Tarp Puncher	19.9 (25.9)	4.5-9.6
Buried, non-tarped	Applicator	90.4 (105)	None	No monitoring	0.7-7.9
Greenhouse, tarped [e]	None	No monitoring	None	No monitoring	0.01-0.07
Handwand					
Replant	Applicator [f]	193 (277)	None	No monitoring	48-60 loc

[a] Data from Tables 13 – 19 in Rotondaro (2004). Handlers were monitored 194 – 810 minutes.
[b] Data from Tables 12 – 17 in Rotondaro (2004). Reentry workers were monitored 240 – 263 minutes.
[c] Chloropicrin concentrations in air (μg/m^3). Multiply values by 0.1487 to express concentrations in parts per billion (see explanation in text). Arithmetic mean estimates rounded to three significant figures. Standard deviation in parentheses beside each mean.
[d] Range of acres treated at 3 sites. Handwand reported number of locations (loc), each of which was a 10 x 10 ft square, corresponding to the small units typically treated when growers replace trees or vines.
[e] Flux monitoring only outside greenhouse applications. No worker exposure monitoring.
[f] Air sampling done in worker breathing zone (reported) and at lower leg. Mean ± SD chloropicrin concentration at lower leg was 235 ± 301 μg/m^3.

In addition to full-shift monitoring, some handlers wore a second air sampler to determine whether elevated chloropicrin concentrations occurred during certain activities such as equipment repair and disconnection of chloropicrin cylinders. The highest concentration detected by these extra pumps was 3,520 μg/m^3, from the driver at Site 13 (monitoring time, 9 minutes). Other than during this task-specific monitoring, handlers monitored by Rotondaro (2004) were exposed to lower chloropicrin concentrations than the highest concentrations reported by Beard *et al.* (1996) during shank broadcast tarped shallow applications: chloropicrin concentrations ranged from 103 to 1,190 μg/m^3 for tractor drivers (mean ± SD = 499 ± 324 μg/m^3) and 213 to 1,330 μg/m^3 for co-pilots (mean ± SD = 626 ± 788 μg/m^3).

The highest reentry chloropicrin concentrations reported by Rotondaro (2004) were for soil shapers following shank broadcast tarped shallow applications (Sites 1 – 3). The arithmetic mean concentration during the 4-hour monitoring period for these workers was 75.9 ± 79.5

$\mu g/m^3$ (range 8.00 – 204 $\mu g/m^3$). Soil shaping follows tarp removal; neither tarp splitting nor tarp removal was monitored by Rotondaro (2004) following broadcast tarped shallow applications. Beard *et al.* (1996) reported chloropicrin concentrations ranging from 69.3 to 4,300 $\mu g/m^3$ for tarp splitters (5 – 6 days post-application; mean $\pm$ SD = 591 $\pm$ 1,080 $\mu g/m^3$) and 6.5 to 3,200 $\mu g/m^3$ for tarp removers (6 – 7 days post-application; mean $\pm$ SD = 626 $\pm$ 788 $\mu g/m^3$) following shank broadcast tarped shallow applications. Examination of monitoring data from both studies suggests that tarp removal following shank broadcast tarped shallow applications is on average the reentry activity with the greatest potential chloropicrin exposure, though the highest individual concentration occurred during tarp splitting.

In addition to the 4-hour monitoring, reentry workers were monitored for the first hour with a second air sampler (Rotondaro, 2004). Results of 1-hour monitoring were not presented in summary tables (the tables show the 4-hour concentrations only), but data from 1-hour monitoring are in Appendix D of Rotondaro (2004). In the text the 1-hour results were summarized as follows: "There were no outstanding differences between the 1 hour and 4 hour data." Examination of 1-hour results shows that, with the exception of Sites 1, 3, 6, and 17 (5 replicates out of a total of 30 monitored), all samples were < LOQ. With the exception of the soil shapers at Site 6, where one of the two 1-hour replicates had a concentration approximately twice that of the corresponding 4-hour replicate (the other 1-hour replicate for that scenario was < LOQ), all of the 1-hour concentrations were within 20% of the corresponding 4-hour concentrations or < LOQ.

Questions About Study

1) Handler tasks covered by monitoring: The times listed for each task are longer than the times given for application. This is in contrast to reentry workers, where task times appear to match monitoring times. The description of driver activities state that they drive to the field, suggesting that monitoring may have begun before any exposure to chloropicrin could have taken place. Do the monitoring times cover times when workers are off-site, or before they begin to handle chloropicrin?

2) Application rates for bedded fields: Beard *et al.* (1996) expressed the application rate as follows: "The target rate was 350 lbs per treated acre. Since the beds reduced the treated acreage by 50% (20" beds/40" on center), the target rate was 175 lbs per gross field acre." Similar information is missing for bedded applications in Rotondaro (2004). Bed sizes are not reported, nor is it stated whether the reported application rate has been adjusted for bedded area.

3) Does CMTF support chloropicrin use to fumigate potting soil? If so, what data are intended to support it? Correspondence from CMTF states that CMTF intended to support this use (Wilhelm, 2001), but DPR disagreed with CMTF's proposal to use data from tree replant handwand applications to cover it (Jones, 2002). This application method was not covered in

the CMTF study (Rotondaro, 2004), nor in the protocol. There may have been some discussion between CMTF and DPR about this topic, as Powell (2002) stated it was unsupported.

cc: Ann Prichard, Senior Environmental Research Scientist (Specialist), Registration Branch

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