

AMMONIA TANK LEAK

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The tank leak that I will describe occurred in January, 1965. The failure was in a new tank and the actual reason for the failure was a 14 in. weld that was missed in the construction of the floor plate. Subsequent hydrostatic tests showed water coming out of the bottom of the tank under the floor and between the floor and the insulation. The belief was that the water was actually trapped under the floor plates and that the weight of the hydrostatic test was squeezing it out. So, it was not considered too seriously. When ammonia subsequently came out, we agreed that there was no ammonia trapped under the floor plates.

Frost spots

The tank was tested and the water was removed. After several feet of ammonia was put into the tank, we started noticing frost spots on the outside wall. In accordance with our other ammonia storage, we were analyzing the vapor space. The vapor space showed high ammonia. We checked the gasket on the manhole; we thought it was leaking there. We are now much more alert to ammonia in the vapor and this may be an important point in these tanks. If you smell ammonia in the vapor space, it could be reasoned that it is coming from the liquid inside the tanks. If the gasket was leaking it would be a pretty good idea to try to seal it. I know some people haven't been able to do it and they've operated successfully. However, we keep the gasket sealed, at least we do now in our present tanks. We analyze the vapor space and sometime we will stop bleeding off the purge in order to accumulate for a period of time just to see how fast vapor concentration builds up. There is always a very minor leak apparently through the gaskets, but you should be able, with nitrogen in the vapor space, to very rapidly reduce this to a low concentration. This is in the insulation between the walls of a double wall tank. Our tanks are both 15,000-ton double wall tanks.

We saw the frost spots so we thought some of that water that was squeezed out of the bottom has picked up some vapor through the manifold and has formed aqua and is conducting heat. To prove this, we just drilled a little hole in the outer shell and we were going to analyze it to show that we got aqua. We had to absorb it in water, it was that strong. In fact, it was pretty close to anhydrous. Once we saw there was anhydrous in the outer shell, then it had to be the flange leak above the manifold or else we had serious trouble. So we checked the flanges and they weren't leaking and we knew that there was a serious problem. It became a problem emptying out the tank.

Emptying an ammonia tank

We called our tank builder, Chicago Bridge and Iron. They came down and talked with us as we emptied the tank. Actually they had some mechanical thoughts on the problem, but to us it was a fairly new idea to empty it. We pumped all of the ammonia out of the tank that we could pump out with a big pump and then started to lose suction on it. Of course, there was still some ammonia in the tank. We didn't want to tear up the big pump. We put a small pump on and pumped all we could out with it. The next step was a little more bizarre, I'm not recommending this, I'm just explaining how we did it. We were kind of feeling our way. We loosened the bolts on the manhole so that if we started getting into trouble we could swing about 2 bolts off and let air in. We very carefully pumped water into the remaining ammonia. We had thermometers all over the tank and were monitoring them, ready to take the manholes off. We do have a vacuum relief on top. We very carefully added a small amount of water and watched the temperatures and nothing too serious happened so we finally added a little more water and finally got enough aqua to analyze. Again, I'm not recommending this, I'm just saying how we did it. During the addition of the water, we added nitrogen as rapidly as possible. We subsequently pumped this out and then purged. We don't generate quite enough pure nitrogen so we purchased a lot of liquid and evaporated it. We purged the tank out with nitrogen and then air, and then went in. The second time we didn't miss the piece that had not been welded the first time. There it was in the tank. It was welded and the tank was again purged with nitrogen and then ammonia vapor and finally filled.

Ammonia carryover

Actually there are a couple of other things on this tank that we might mention here. We had a problem with carryover in our particular tank. We had a design on the top which I'm not quite sure why it was used. It was called a mist separator bottle. We had a 120-ft. tank as a separator, but that didn't look good enough so the tank had about a 3-ft. separator built on top. It didn't work. We had carryover mist and the problem was that the vent from the separator at the top of the tank wasn't big enough. The tank would pressure up due to flashing of ammonia and that would hold the liquid up in the separator and it carried over into our line. Our compressor was about half a mile away uphill in the compressor house so it would fill a seal leg in the vapor line with ammonia. Since the tank operates

at 7 in. and the seal leg was greater than 7in., the vapor only had one other place to go and that was out through the relief valves. That has been corrected.

I think like most, we do not have an infinite time to check everything so these things are going to come up. They will keep coming up and we mention them just so that in building your installation, you can kind of watch for them. Looking back, we were going to put a big vaporization pot there and then we thought it might be smart just to try to stop the mist from coming over. I think we've done that by enlarging the vapor line between the pot and the top of the tank.

Airplane trouble

We had another interesting experience. One Sunday morning, we had a dentist and another fellow up in a small airplane flying around when the carburetor froze up. They were looking for an airstrip and the best thing they could see was the blacktop road in our plant leading straight to a low pressure tank. They weren't too good, they missed that strip and hit a field right along side of it. I gave them a pretty hard time getting the airplane out, I made them get special equipment to pull it out. I made them uncomfortable because I didn't want anybody else using the plant for an emergency landing field. He was heading right for our low pressure tank when he finally got it stopped.

Nitric acid vent

We had another interesting experience on this tank, our nitric acid vent from our nitric acid plant blew over onto our vapor relief valve. We had a nylon seal impregnated with a Teflon on the valve. We have a rule, which we adapted from something I think someone had mentioned earlier, to go around and check the relief valves, to make sure that they are working. This one we found wasn't working, it was cemented down. Apparently the fumes from the nitric acid plant had attacked this seal and caused it to cement on to the seat. We raised the pressure to about 10 in. to see if that would lift and it wouldn't. That is about as far as we went trying this test. We went up with maintenance men and, here again confirming what others have said, we have had no serious difficulty in pulling the pressure down to about 1/2 in. positive. We put men in chemical suits with fresh air masks and had them go in and take off this relief valve. It isn't easy and you don't want any inexperienced people doing it. We took the valve and assembly off. We have taken the varac housing clean off and haven't had any great problem. When you start wondering if that valve is going to work right or if the seats are bad, there are only two things to do and they are either empty the tank out or take the valve off. You don't have any other choices.

These were not serious things, they are something you catch fairly quick if you keep your tanks inspected and I am sure you all do. They are just a series of small problems and I am sure most of you could relate similar ones.

DISCUSSION

WARREN—Dupont: The source of the following desires to remain anonymous. I will read what I have for your benefit:

"This accident happened in January, 1964, at 11:10 a.m.. An anhydrous ammonia tank truck was connected to a storage tank for unloading with a 2-in. rubber hose, and a 3/4-in. rubber equalizing hose from the tank truck compressor. This work was done by the transport company's truck drivers. Only the couplings were supervised by our pilot plant superintendent. The hoses were furnished by the truck company. Almost immediately as the truck operated compressor was en-

gaged, the hose ruptured, apparently at tank truck pressure, approximately 170 lb./sq. in. gauge. Two men were seriously injured with liquid ammonia and ammonia vapors. Two additional men received injuries from the vapors. All were taken to the hospital by ambulance. The nature of the injuries were severe chemical burn damage to the trachea and upper respiratory systems from ammonia vapor inhalation. Exterior chemical burns caused the death of two men critically injured. A visual inspection and thorough test indicate a defective hose was the apparent cause of the rupture. Action taken: in the future the plant will furnish its own hose and will provide proper care and rigid inspection prior to its use."