

Claims

1. A process for separating anhydrous methanesulfonic acid from a reaction mixture comprising methanesulfonic acid and sulfuric acid by distillation with at least three functional steps, characterized that in the first functional step the reaction mixture is fed into a first column K1, where the top stream of column K1 is then transferred to the second functional step, wherein this second functional step is a second column K2, in which purified anhydrous methanesulfonic acid is obtained from its bottom stream, and wherein the bottom stream of column K1 is transferred to a third functional step, wherein this third functional step is either a third column K3 or an evaporator W3-1, in which the top stream is recycled back into column K1, and in which the bottom stream is separated from the distillation process.
2. A process according to claim 1, in which in addition to evaporator W3-1 further (n-1) evaporators W3-2, W3-3, ... , W3-n, n being an integer from 2 to 10, are used in the third functional step, wherein the bottom stream of each evaporator W3-k, k being an integer from 1 to (n-1)) is transferred to each evaporator W3-(k+1), and wherein the top stream of each evaporator W3-(k+1) is transferred back to each evaporator W3-k, while the bottom stream of evaporator W3-n is separated from the distillation process.
3. A process according to claim 1 or 2, wherein the reaction mixture comprises 55 to 95 wt.-% methanesulfonic acid, 5 to 45 wt.-% sulfuric acid, 0 to 5 wt.-% methanesulfonic acid anhydride, 0 to 1 wt.-% side products, 0 to 5% SO<sub>3</sub>, 0 to 5 wt.-% of water and traces of methane, wherein the sum of all components sums up to 100 wt.-%, and wherein the side products comprise methylmethanesulfonate and/or methylbisulfate and/or methanedisulfonic acid, and wherein the amount of components other than methanesulfonic acid and sulfuric acid is in the range of 0 to 10 wt.-%.
4. A process according to claim 1, wherein the at least two columns K1, K2 and optionally K3 can each be set-up as one column K1 and K2 and optionally K3, or as several columns with same functionalities K1, K2 and optionally K3 operated in parallel.
5. A process according to claims 1 to 4, wherein the bottom residue of column K3 or evaporator or cascade of evaporators W3-n contains at least 60 wt.-% of sulfuric acid.
6. A process according to claims 1 to 5, wherein the pressure at the head of the columns and optionally evaporators ranges from 0.1 to 50 mbar, for all distillation columns K1, K2 and optionally K3 and evaporators W3-n.

7. A process according to claims 1 to 6, wherein the temperature at the bottom of the columns and optionally evaporators ranges from 140 to 220°C, in all distillation columns K1, K2 and optionally K3 and evaporators W3-n.
- 5 8. A process according to claims 1 to 7, wherein purified MSA leaves the distillation at bottom of K2 with a specification of < 500 ppm sulfuric acid, < 1000 ppm MMS, 0.5 wt.-% water.
- 10 9. A process according to claims 1 to 8, wherein MSA leaves the distillation with a purity of at least 98 wt-%.
10. A process according to claims 1 to 9, wherein the process is a batch or a continuous process.
- 15 11. A process according to claims 1 to 10, wherein the MSA recovery rate in the distillation is at least 80%.
- 20 12. A process according to any of the preceding claims 1 to 11, wherein at least a part of the bottom fraction of the MSA distillation column K3 is not purged from the system but at least partially recycled to the synthesis step of the starter solution or to the synthesis step of the MSA synthesis with methane and SO<sub>3</sub>.
- 25 13. A process according to any of the preceding claims 1 to 12, wherein the residence time in each distillation column and optionally evaporators W3-n is below 5h.
- 30 14. A process according to any of the preceding claims 1 to 13, wherein the ratio of the inner column diameter in the sump and the inner column diameter of the column directly above the sump for each column is in the range from 0.20 to 0.99.

Recovery of water-free methanesulfonic acid from the bottom stream of a distillation column

Abstract

- 5 Process for separating anhydrous methanesulfonic acid from a reaction mixture comprising methanesulfonic acid and sulfuric acid by distillation with at least three functional steps and the use of said methanesulfonic acid.